ROYAL AEROSPACE ESTABLISHMENT

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CRAG TEST METHODS FOR THE MEASUREMENT OF THE ENGINEERING PROPERTIES OF FIBRE REINFORCED PLASTICS

edited by

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Procurement Executive, Ministry of Defence
Farnborough, Hants

UNLIMITED

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SUMMARY

This Report is the third and final issue of a document describing test methods suitable for the measurement of the engineering properties and other associated properties of fibre reinforced plastics. Specimen configurations and testing procedures are detailed and the applicability of the tests to the different types of fibre reinforced plastics is discussed.

Departmental Reference: Materials & Structures 218

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LIST OF CONTENTS

BACKGROUND AND MEMBERSHIP OF THE CRAG WORKING GROUP ON TEST METHODS

The Working Group was inaugurated in 1980 by the Composite Research Advisory Group (CRAG) with terms of reference including a requirement to survey and rationalise current test procedures for fibrous composite materials. This group has now agreed test methods for the generation of materials design data, primarily on undirectional composite materials for use in design aids such as laminated plate theory, but also on multidirectional fibre composites where this is not obtainable by other means. This Report is the third and final formal issue of test methods by the group, which has now been dissolved. The methods detailed under sections 301 and 800 - 1001 are new additions in this document. Other methods have been revised where appropriate to cover developments since their previous issue as RAE reports TR 84102 and TR 85099.

The test methods which follow are a result of collaboration between the Royal Aircraft Establishment, Farnborough, British Aerospace plc (Warton, Woodford and Stevenage Divisions), Westland Helicopters Limited, Short Brothers, Rolls Royce and Ciba Geigy (UK) Limited.

Current membership of the group:-

Mr N.L. Bottrell	-	WHL Yeovil (Chairman)
Mr D. Bradwell	-	BAe Stevenage
Mr P.E. Brooks	-	Ciba Geigy (UK) Ltd (Secretary)
Mr H. Calder	-	BAe Woodford
Dr P.T. Curtis	-	RAE Farnborough (MS4)
Mr E.C. Edge	-	BAe Warton
Mr A.B. Hamill	-	Short Bros, Belfast
Dr J. Hill	-	Rolls Royce, Derby

0 INTRODUCTION

Scope

The test methods recommended in these data sheets apply to resin matrix composites reinforced with orientated continuous fibres. They are valid for all grades of carbon glass and aramid fibres, in both U/D tape and woven fabric forms including mixed fibre hybrid combinations, provided that the modes of failure are representative. Epoxy resin matrix systems were used in the development of these methods, but, although confirmatory test evidence is lacking, other resin systems should be equally suitable provided that the composite properties fall within any prescribed test limitations and that valid failure modes are obtained.

All multidirectional laminates, including woven fabric laminates, must have their ply orientations and stacking sequence balanced about the mid-plane and the longitudinal axis to ensure axial loading and to prevent rotational, flexural, or off-axis displacements. GREAT BRITAIN. (JIS)

All laminates should be assessed for quality using an ultrasonic scanning technique, as described in section 1001.

Machining

The machining of fibre reinforced plastics must be carried out with care to minimise damage, *eg* delamination, splitting, etc. CFRP and GRP can be machined with relative ease but KRP is more difficult and reference should be made to 'Machining of Kevlar Composites' which is obtainable from Du Pont deNemours International SA, Industrial New Products Section, Textile Fibres, PO Box CH-1211, Geneva 24.

CFRP and GRP may be readily sawn or drilled but require the use of diamond impregnated or tungsten carbide tools because of the rapid dulling of cutting surfaces. All tools must be kept clean and free from grit. High pressure water jets have also proved successful in the cutting of these materials.

The drilling of holes requires particular care to ensure that delamination does not occur on the exit face when the drill breaks through. The composite should be adequately backed by a suitable piece of sacrificial material and feed rates should be slow but uniform.

Saw marks on the edges of test specimens may be detrimental to the results obtained and should be removed. Any such marks on the longitudinal edges may be removed by grinding or abrading with suitable (320 grade approximately) wet/dry emery paper.

Provided the moulded thickness is correct, laminated surface finishes are generally acceptable. It should be noted that many peel plies used in the manufacture of composite materials can give rise to a resin rich dimpled surface which may result in the overestimation of panel thickness. This effect may be particularly significant for thin specimens and flexure tests, when removal of the dimples may be advisable. If a specimen has to be reduced in thickness (unidirectional laminates only) surface grinding may be used.

Grinding operations for CFRP and GRP should be undertaken using a diamond coated or open grit carbide grinding wheel. Cutting speeds should be adjusted to avoid over-heating of the specimen. Certain proprietary liquid coolants may be used to prevent free dust and minimise wheel clogging. It is essential to ensure that any coolant used does not affect the matrix properties and can be cleaned off prior to attaching end tabs.

When any form of dry machining is undertaken it is essential that an efficient dust extraction system is employed.

Furthermore, personnel involved should wear the appropriate safety equipment, *eg* filter masks.

Adhesive bonding

Thorough surface preparation is essential prior to bonding on end fittings. Test pieces and end tags should be abraded and degreased to remove any surface contaminants in the area to be bonded. Dimpled surfaces produced by a peel ply during manufacture may only require degreasing. Aluminium alloy parts should be etched and cleaned in accordance with Method 0 of BSI Code of Practice CP3012 (Method 0 of DEF Standard 03-2/1).

The adhesive employed must be capable of withstanding the particular test environment, but attention must be paid to the residual thermal stresses arising from any hot bonding, in particular the peeling forces at the ends of the laminates. Cold cure epoxy adhesives are recommended for normal applications. Hot cure epoxy films and GRP end tags (to reduce the residual stresses) are necessary for severe test environments.

Test machines and instrumentation

The test machine should conform to the calibration standard BS 1610: 1985 Grade 1.0 and be equipped to record applied load and cross-head deflection rate.

Accurate load alignment and cross head movement are essential for satisfactory testing. If necessary a suitable test rig should be employed to ensure the required accuracy.

Continuous or 'ramp' loading is preferred with continuous load and strain recording. If incremented loading is used it should be noted in the test report.

Strain gauges are recommended rather than extensometers for strain recording, as their results have been found to be more reliable. It is recommended that strain gauges are attached to both faces of the specimens to enable any out-of-plane bending effects to be measured. Careful surface preparation is necessary before bonding on the gauges. The gauges should be given a suitable protective covering if the specimen is to be subjected to environmental conditioning.

Strain gauges can sometimes fail during prolonged fatigue testing, and it is therefore prudent to undertake a secondary check at intervals, possibly by monitoring machine ram displacements.

Report

The report for any test performed should include:

(i) A description of the test method and type of specimen used, together with its critical dimensions, type of end fittings and adhesives employed.

(ii) Fibre and resin type, weave style, preimpregnate batch identification, number of plies and their orientation and stacking sequence, laminate processing details.

(iii) The fibre volume fraction of the laminate as described in section 1000.

(iv) The environmental history of the specimen prior to test.

(v) The environmental conditions of temperature and humidity versus time during test.

(vi) Variations in the specimen average moisture levels throughout manufacture conditioning, and testing as monitored by the traveller.

(vii) The number of specimens tested for each property. (A minimum of five is recommended for static testing.)

(viii) The failure load, stress and mode of failure (if applicable) for each specimen, together with the mean failure stress for all the specimens and the coefficient of variation. (Coefficient of variation is given by:

$$C_v = \frac{100}{\bar{x}}\left[\frac{\Sigma(x - \bar{x})^2}{n - 1}\right]^{\frac{1}{2}}$$

where $\bar{x}$ is the arithmetic mean and n is the number of values of x .)

(ix) Actual and nominal thickness deduced from nominal cured ply thickness at a specified fibre volume fraction.

(x) Where applicable - the stress - strain curve, secant modulus and Poisson's ratio for each specimen, together with the mean secant modulus (and strain range used) and Poisson's ratio for all the specimens and the coefficient of variation.

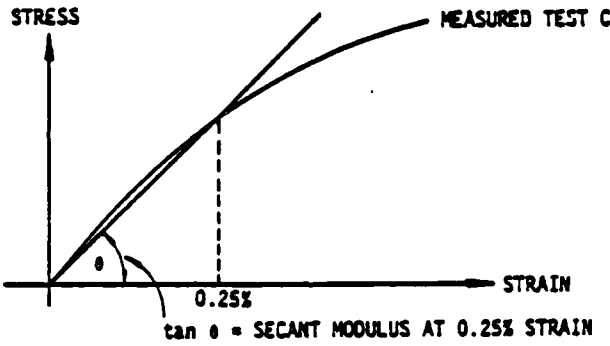


Fig 0.1 Typical stress-strain curve

NOTES: (1) A strain range of 0.25% is recommended, but this may be varied if necessary to suit a particular application.

(2) Any initial irregularities in the stress/strain curve should be ignored and the curve should be extrapolated back to determine the origin.

(3) The shape of the curve may vary with material and test.

(xi) For fatigue tests - mean stress, stress amplitude, number of stress cycles to failure, frequency of loading, or load spectrum, and if appropriate the residual strength and/or stiffness.

(xii) The date of the test.

(xiii) Any observations made during the test that may be relevant to the results obtained or any deviations from the recommendations, *eg* incremental instead of continuous loading.

Deviations from the standards

In certain cases some dimensional variations can be permitted without significantly affecting the validity of the test results. Where applicable these are given in the appropriate Data Sheets. Any deviations outside the permitted range may invalidate the test results.

Specific comments

(i) Multidirectional tensile specimen (Data Sheets 303 and 402) Notch sensitivity varies with hole size, particularly for GRP.

(ii) The maximum testing times recommended may be too long to ensure the absence of creep deformation particularly at elevated temperature and with high moisture content. In all cases a time to failure at the lower end of the range is preferred.

100 METHOD OF TEST FOR INTERLAMINAR SHEAR STRENGTH OF FIBRE REINFORCED PLASTICS

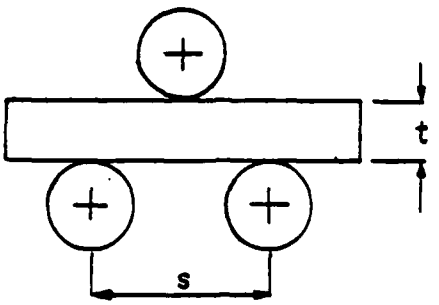


Fig 100.1

Description

This is a three-point flexure test for reinforced plastic specimens with a span to depth ratio low enough to produce matrix shear failure.

Materials

The test may be used for any form of material, subject to the 'validity' requirements below. The test specimen must be axially orthotropic.

Nomenclature

t = specimen thickness	w = specimen width
t_n = nominal specimen thickness	L = specimen length
S = span between supports	P = load at failure

Dimensions

t_n = 2.0 mm (nominal or nearest mouldable thickness)	$w = 5t_n \pm 0.25$ mm
$L = (5t_n + 10.0 \text{ mm}) \pm 0.5$ mm	$S = 5t \pm 0.5$ mm (1) $S = 4t \pm 0.5$ mm (2)

All dimensions must be parallel to ±0.05 mm

Support rollers diameter 6 mm

Loading roller diameter 6 mm (10 mm acceptable).

NOTES: (1) For materials having a flexural strength to shear strength ratio greater than 10:1.

(2) For materials having a flexural strength to shear strength ratio less than 10:1.

012

Test requirements

The specimen must be located symmetrically on the support rollers. The loading roller must be constrained to move vertically above the centreline of the specimen. A jig shall be used to ensure accurate alignment.

Load shall be increased uniformly to cause failure within 15-45 seconds.

Calculation

Interlaminar shear strength is given by:

$$ILSS = 0.75\ P/wt$$

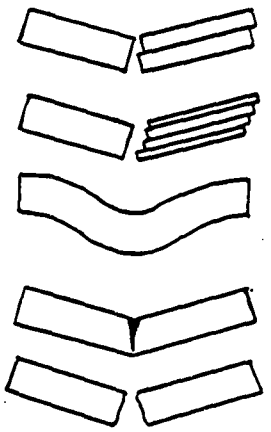
Accurately measured values of w and t must be used. Adjustment of the result to a standard volume fraction (Vf) is not permissible. Report ILS strength, Vf .

It is important to note that the value obtained for ILSS is highly dependent upon the span to depth ratio.

Validity

For result to be meaningful, the failure mode must be either single or multiple shear or plastic deformation with evidence of shear failure.

Flexural failure or plastic deformation without evidence of shear failure will produce only a lower bound value of ILSS.



single shear - VALID

multiple shear - VALID

Plastic deformation - VALID
with evidence of
shear failure

Flexural failure - INVALID

Fig 100.2

101 METHOD OF TEST FOR IN-PLANE SHEAR STRENGTH AND MODULUS OF FIBRE REINFORCED PLASTICS

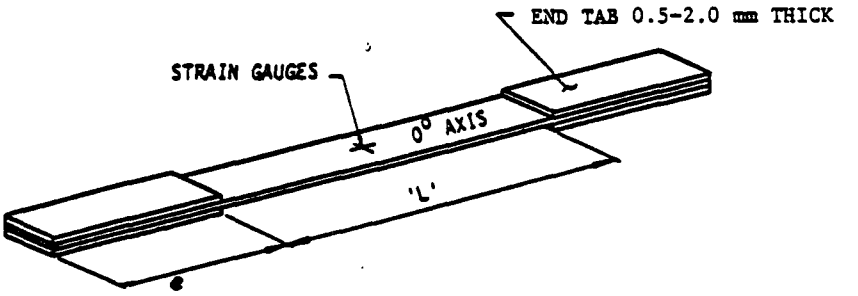


Fig 101.1

Description

This specimen is used to determine the in-plane shear strength and modulus of reinforced plastic materials. The test is suitable for either unidirectional tape or woven fabric, and measures the shear properties associated with an individual layer. The specimen, which is subjected to tensile loading, comprises a laminate with the fibres inclined at 45 degrees and -45 degrees to the longitudinal axis. To avoid distortions and induced bending the lay-up must be fully symmetric. In the case of undirectional tape, the layers are alternately at 45 degrees and -45 degrees up to mid-plane, then alternately at -45 degrees and 45 degrees, with an equal number of layers in each direction.

A pair of strain gauges (at 0 degrees and 90 degrees to 0 degree axis) is required at the centre of the specimen to measure modulus. (It is preferable to use a pair on each face to average out specimen bending.)

The end tabs are either soft aluminium alloy or GRP attached using a suitable adhesive.

Testing without end tabs is permissible provided that suitable end grips are used.

Nomenclature

e = length of end tabs

t = measured thickness of specimen

w = measured width of specimen

L = free length

P_u = load at failure

ε_1 , ε_2 = strains recorded by 0 degree and 90 degree gauges at load P .

Dimensions

e = 50 mm minimum

t = 2.0 mm (nominal or nearest mouldable thickness - see also 'Description'

w = 25.0 mm

L = 100 mm (minimum)

Tolerances

The specimen must be flat and the end tags parallel and aligned to within ±0.05 mm.

Permitted deviations from standard.

Tolerance on width = ±0.25 mm

Tolerance on length = ±1 mm.

In exceptional circumstances w may be reduced to a minimum of 20 mm, and L may be reduced to a minimum of 50 mm.

Test requirements

Specimen must be carefully aligned in test machine jaws to avoid induced specimen bending. Ideally tensile load (or strain) should be increased uniformly to cause failure within 30-60 seconds. However this may not be feasible for many toughened composites which can fail at strains in excess of 8-10%. In this case the loading (or strain) rate should be selected so that at least 90% of the anticipated failure load is applied in the range 30-60 seconds. For modulus determination strain versus load must be recorded separately for 0 degree and 90 degree gauges. Care should be taken to ensure adequate and reliable bonding between the coupon and strain gauges (if used), particularly when testing in hot/wet environments.

Calculations

Shear strength is given by $\tau_u = 0.5\ P_u/wt$

Shear stress at load P is given by $\tau_{12} = 0.5\ P/wt$

Shear strain at load P is given by $\gamma_{12} = \varepsilon_1 - \varepsilon_2$.

Since stress versus strain graph may be nonlinear, shear modulus is defined as:

G_{12} = secant modulus at 0.5% shear strain (1) .

Report shear strength and modulus, stress versus strain curve to failure, actual moulded thickness, nominal thickness V_f .

NOTES: (1) For method of derivation see Part 0 - Introduction.

(2) Creep may occur at high stress levels, particularly at elevated temperature, rendering the strain values time dependent.

(3) Rail shear test (Ref ARC CP1381) may be used as an alternative for shear strength measurement only.

102 METHOD OF TEST FOR LAP SHEAR STRENGTH OF FIBRE REINFORCED PLASTICS

Description

These specimens are used for comparative tests on adhesives used for bonding laminated composite materials, and not to obtain design data on adhesive properties nor strength data for specific applications (see Note). The single lap specimen A would normally be used for quality control tests, whilst either specimen A or the double lap specimen B may be used for comparative testing, *eg*

(i) to compare the adhesion shear strength obtained with different adhesives;

(ii) to study the influence of surface preparation;

(iii) to study the influence of fibre orientation in the surface layers at the bonded surfaces;

(iv) to study the influence of environmental effects.

Specimen B would normally be expected to give the higher average shear stress at failure because of inherently lower peel stresses at the ends of the joint.

In each series of comparative tests, only the parameter under investigation should be varied, all other parameters remaining constant throughout.

Note that the size and quality of the adhesive fillet can affect the test result.

The adherends may be made from unidirectional tape or woven material provided the laminate is axially orthotropic to obviate induced bending.

The specimens are illustrated in Fig 102.1

NOTE: For the derivation of design data on the cohesive properties of adhesives, the test method given in Draft European Standard Aerospace Series pr EN 2243-6 should be used.

The average shear stress in a joint at failure is a function of the overlap length, the stiffness of the adherends, the stress versus strain characteristics of the adhesive and the level of peel stresses at the ends of the joint. Therefore, to obtain design strength data for a specific application, a fully representative test specimen must be used.

Nomenclature

t = thickness of adherends

t_a = thickness of adhesive

w = width of specimen

l = overlap length of joint

P = load at failure

τ_{ave} = mean shear stress in joint at failure.

Dimensions

t = 2.0 to 2.5 mm, depending on laminate configuration

w = 25 mm

l = 12.5 mm.

Permitted deviations from the standard

For particular purposes:

t may be between 1.2 and 3.0 mm

w may be between 17 and 30 mm but not less than 10t.

Tolerances

All edges must be square and the sides of the specimens must be parallel to within ±0.05 mm.

The adherends must be flat and of uniform thickness to within ±0.05 mm.

The adherends must be parallel with a uniform thickness of adhesive to within ±0.025 mm.

Overlap length shall be accurate to ±0.25 mm.

Ideally it is recommended that the moisture content of the adherends be kept as low as possible.

Test requirements

The test specimen must be carefully aligned in the jaws of the test machine to avoid induced bending.

Tensile load should be increased uniformly to cause failure within 30-90 seconds.

When environmental conditioning is required it should be undertaken on the complete specimen after manufacture.

Calculations

The mean shear stress at failure is given by:

$$\tau_{ave} = P/wl \quad \text{(specimen A)}$$

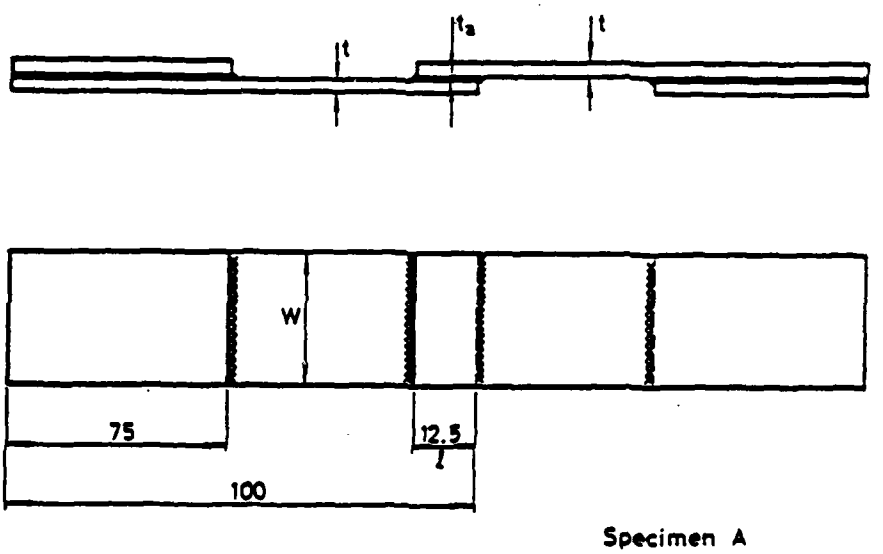
$$\tau_{ave} = P/2wl \quad \text{(specimen B)}$$

accurately measured values of w and l must be used.

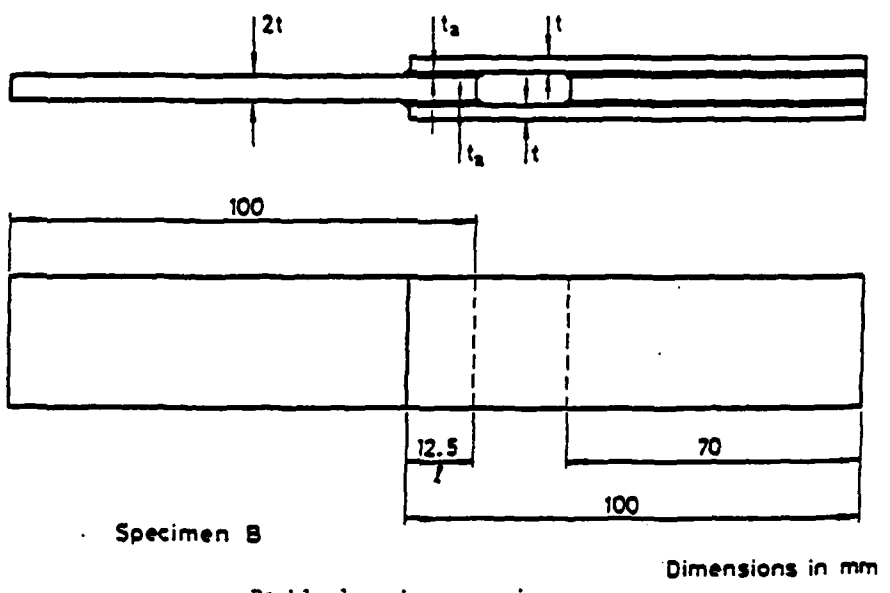
Report mean shear stress at failure, mode of failure (cohesive adhesive, or delamination), adhesive type, measured bond line thickness, laminate configuration including orientation of surface layers, and surface preparation of adherends.

Validity

All shear modes of failure are valid results. (The maximum achievable shear stress will be obtained with cohesive failure within the adhesive.)



Single lap shear specimen



Double lap shear specimen

Fig 102.1 Adhesive test specimens

200 METHOD OF TEST FOR FLEXURAL STRENGTH AND MODULUS OF FIBRE REINFORCED PLASTICS

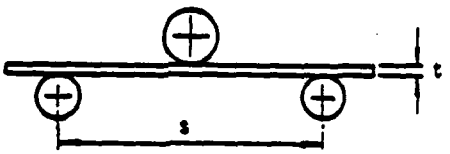


Fig 200.1

Description

This is a three-point flexure test for reinforced plastic specimens with span to depth ratio high enough to produce bending failure.

Materials

The test may be used for either unidirectional or woven material. The material direction under investigation must be orientated along the specimen length.

Nomenclature

t = specimen thickness

w = specimen width

S = span between supports known to ±0.2%

P = load at failure

m = slope of linear load/ deflection graph (N/mm)

Dimensions

t = 2.0 mm (or nearest mouldable thickness)

w = 10.0 mm ±0.25 mm

Table of Span Requirements

Composite reinforcement	Alignment of fibres to beam axis	S/t	
Unidirectional carbon fibre	0 degree	40/1	±1 mm
Unidirectional carbon fibre	90 degrees	25/1	
Woven carbon fibre	0 degree/90 degrees	25/1	
Unidirectional glass fibre	0 degree	20/1	
Unidirectional glass fibre	90 degrees	20/1	
Woven glass fibre	0 degree/90 degrees	20/1	
Woven aramid fibre	0 degree/90 degrees	16/1	

Minimum overhang of specimen beyond rollers 5.0 mm
Support rollers 10 mm diameter
Loading roller 25 mm diameter
Loading roller to be located at mid-span within ±0.5 mm.

The diameter of the support rollers may be reduced to a minimum of 6 mm.

The diameter of the loading roller may be reduced to a minimum of 10 mm.

(The larger diameter rollers are recommended to prevent specimen indentation.)

Tolerances

Width and thickness shall be uniform to within 0.05 mm.

Test requirements

A jig should be used to ensure accurate alignment. Load should be increased uniformly to cause failure within 30-180 seconds. Central deflection versus load must be recorded if flexural modulus values are required.

Calculations

Flexural strength is given by:

$$f_F = 1.5\ PS/wt^2 \quad .$$

Flexural modulus is given by

$$E_F = s^3m/4wt^3 \quad .$$

Values of w and t must be obtained by accurate measurement at the middle of the specimen.

Report flexural strength and modulus, Vf , nominal and actual thickness.

Validity

The flexural test is primarily for material control purposes and will not provide reliable tension or compression data. The test results will depend upon the actual fibre volume fraction (Vf) of the laminate.

300 METHOD OF TEST FOR THE LONGITUDINAL TENSILE STRENGTH AND MODULUS OF UNIDIRECTIONAL FIBRE REINFORCED PLASTICS

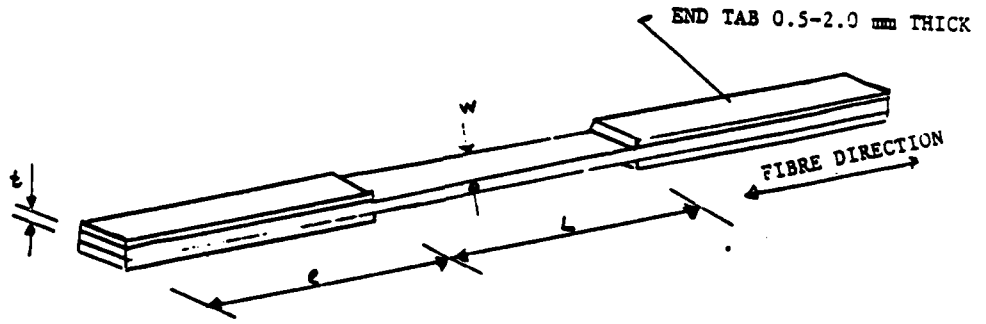


Fig 300.1

Description

This specimen is used to determine longitudinal tensile strength and modulus for unidirectional laminates.

The use of end tabs is recommended to inhibit splitting of the specimen. For moist or hot conditions GRP end tabs are recommended. The curing temperature of the glass fibre and the adhesive used to bond the tabs on to both specimen faces should be suitable for use at the required test conditions. For testing under dry ambient conditions soft aluminium alloy end tabs are satisfactory.

Nomenclature

- t = measured thickness of specimen
- w = measured specimen width
- L = free length
- e = length of end tabs (or gripped length)
- P = load at failure
- f_{1T} = longitudinal tensile strength
- E_{1T} = secant modulus
- ν = Poisson's ratio.

Dimensions

t = 1.0 mm* (nearest mouldable thickness)

w = 10-20 mm

L = 100-150 mm

e = 50 mm minimum.

* Tolerances

Width and thickness must be uniform to ±0.04 mm.

Specimen profiles including end tabs must be symmetric about longitudinal axis to ±0.05 mm.

Fibre alignment must be parallel with the specimen longitudinal axis within 0^{o} 30'.

Test requirements

Specimens must be carefully aligned in test machine jaws to avoid induced specimen bending. Tensile load (or strain) should be increased uniformly to cause failure within 30-90 seconds. For modulus determination axial strain versus load must be recorded. (Transverse strain measurements are also required if Poisson's ratio is to be determined.)

Calculations

Longitudinal tensile strength is given by $f_{1T} = \frac{P}{wt}$.

Accurately measured values of w and t must be used.

The stress versus strain graph is frequently nonlinear, with modulus changing slowly with increasing strain. It may be required to use all the data generated right up to failure, *eg* for laminate strength prediction. Usually, however, a spot value is specified for general design use, longitudinal tensile modulus being defined as either:

E_{1T} = secant modulus at S% longitudinal strain

or

$$E_{1T} = \frac{100 \text{ (stress at S1\% long. strain - stress at S2\% long. strain)}}{S_1 - S_2} .$$

Similarly Poisson's ratio may be defined as either:

$$\nu = \frac{\text{transverse strain}}{\text{longitudinal strain}} \text{ at S\% longitudinal strain}$$

or

$$\nu = \frac{100(\text{trans strain at S1\% long strain} - \text{trans strain at S2\% long strain})}{S_1 - S_2} .$$

The values of S, S_1 and S_2 can vary with material characteristics and project requirements. In the absence of such pointers, it is recommended that the simpler definition is followed with $S = 0.25$, as described in the Introduction section 0.

NOTE: The properties derived will be approximately proportional to the fibre volume fraction of the laminate.

Report longitudinal tensile strength and modulus, stress versus strain curve to failure, Poisson's ratio, V_f, nominal and actual thickness.

Validity

To be valid for design data, failure must occur in the central region.

301 METHOD OF TEST FOR THE TRANSVERSE TENSILE STRENGTH AND MODULUS OF UNIDIRECTIONAL FIBRE REINFORCED PLASTICS

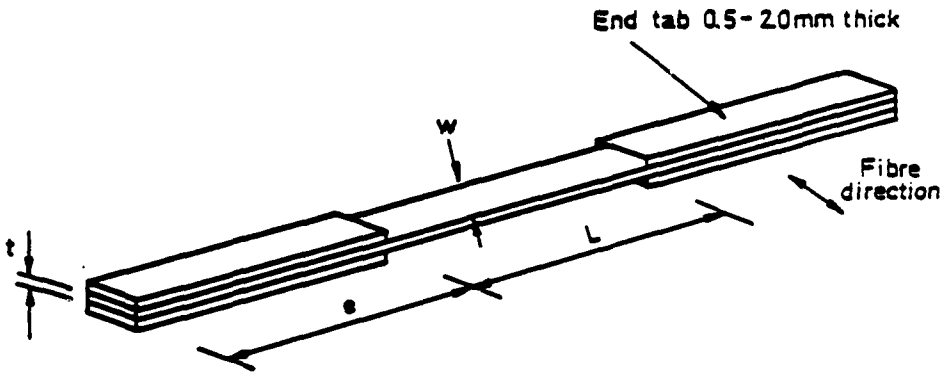


Fig 301.1

Description

This specimen is used to determine transverse tensile strength and modulus for unidirectional laminates, *ie* the specimens will have all the fibres oriented at 90 degrees w.r.t the loading direction.

The use of end tabs is recommended to inhibit splitting of the specimen. For moist or hot conditions GRP end tabs are recommended. The curing temperature of the glass fibre and the adhesive used to bond the tabs onto both specimen faces should be suitable for use at the required test conditions. For testing under dry ambient conditions soft aluminium alloy end tabs are satisfactory.

Nomenclature

- t = measured thickness of specimen
- w = measured specimen width
- L = free length
- e = length of end tabs (or gripped length)
- P = load at failure
- f_{2T} = transverse tensile strength
- E_{2T} = secant modulus
- ν = Poisson's ratio.

Dimensions

t = 2.00 mm* (nearest mouldable thickness)

w = 10-20 mm

L = 100-150 mm

e = 50 mm minimum.

* Tolerances

Width and thickness must be uniform to ±0.04 mm.

Specimen profiles including end tabs must be symmetric about longitudinal axis to ±0.05 mm.

Fibre alignment must be parallel with the specimen longitudinal axis within 0° 30'.

Test requirements

Care should be taken when cutting blank coupons from the panel to minimise edge damage induced by cutting, since this can influence the measured strengths.

Specimens must be carefully aligned in test machine jaws to avoid induced specimen bending. Tensile load (or strain) should be increased uniformly to cause failure within 30-90 seconds. For modulus determination axial strain versus load must be recorded. (Longitudinal strain measurements are also required if Poisson's ratio is to be determined.)

Calculations

Transverse tensile strength is given by $f_{2T} = \frac{P}{wt}$.

Accurately measured values of w and t must be used.

Since the stress versus strain graph is frequently nonlinear, the transverse tensile modulus is defined as either:

E_{2T} = secant modulus at S% transverse strain

or

$$E_{2T} = \frac{100 \text{ (stress at } S_1\% \text{ trans. strain - stress at } S_2\% \text{ trans. strain)}}{S_1 - S_2} .$$

Similarly Poisson's ratio may be defined as either

$$\nu = \frac{\text{longitudinal strain}}{\text{transverse strain}} \text{ at S\% trans strain}$$

or

$$\nu = \frac{100(\text{long. strain at } S_1\% \text{ trans. strain} - \text{long. strain at } S_2\% \text{ trans. strain})}{S_1 - S_2}$$

The values of S, S_1 and S_2 can vary with material characteristics and project requirements. In the absence of such pointers, it is recommended that the simpler definition is followed with $S = 0.25$, as described in the Introduction section 0.

Report transverse tensile strength and modulus, stress versus strain curve to failure, Poisson's ratio, V_f.

Validity

To be valid for design data, failure must occur in the central region.

302 METHOD OF TEST FOR THE TENSILE STRENGTH AND MODULUS OF MULTIDIRECTIONAL FIBRE REINFORCED PLASTICS

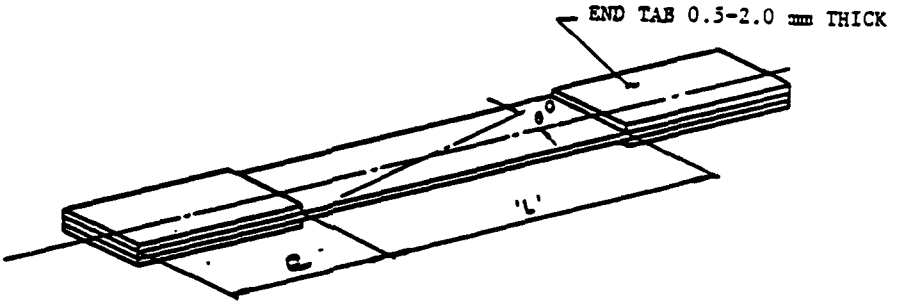


Fig 302.1

Description

This specimen is used to determine the tensile strength and modulus of multidirectional laminates (unnotched). The test may be used for either unidirectional tape or woven materials, provided the laminate is axially orthotropic to obviate induced bending.

In the angled plies, no individual fibres should run under the tabs at both ends of the specimen. The free length is chosen so that a non-axial fibre can run across the full width of the specimen and be at least half its specimen width short of the end tabs at each end, *ie* minimum $L = W\left(1 + \frac{1}{\tan \theta}\right)$.

For testing under dry ambient conditions soft aluminium alloy or GRP end tabs are suitable. For moist or hot conditions GRP end tabs are recommended. The tabs are attached using an adhesive suitable for the test environment.

The end tab adhesive shear strength will limit the level of load input and hence limit the amount of 0° fibres present in the laminate, *eg* typically for tests under dry ambient conditions a maximum thickness of 1.5 mm of 0° carbon fibres can be allowed.

In exceptional circumstances testing without end tabs may be permissible provided that suitable end grips are used.

Nomenclature

e = length of end tabs

t = measured thickness of specimen

w = measured width of specimen

L = free length

θ = angle between fibres and longitudinal axis

P = load at failure.

Dimensions

e = 50 mm minimum

w = 9t minimum, typically 10t (absolute minimum = 20 mm)

t = 1.0 to 4.0 mm depending on laminate configuration

L not less than $w(1 + 1/\tan \theta)$ or 100 ±1 mm whichever is greater.

Tolerances

Edges of specimen must be parallel to ±0.1 mm. Specimen must be flat and end tab faces parallel and aligned to within ±0.05 mm.

For small angles of $\theta(<15^{\circ})$ L may be reduced to $w/\tan \theta$.

Test requirements

Specimen must be carefully aligned in test machine jaws to avoid induced specimen bending. Tensile load (or strain) shall be increased uniformly to cause failure within 30-90 seconds. For modulus determination strain versus load must be recorded.

Calculations

Tensile strength for plain specimen is given by:

$$f_T = \frac{P}{wt} .$$

Since stress versus strain graph may be nonlinear, tensile modulus is defined as:

E_T = secant modulus at 0.25% axial strain (1)

Poisson's ratio = $\frac{\text{transverse strain}}{\text{axial strain}}$ at 0.25% axial strain (1) .

012

NOTE: (1) For method of derivation see Introduction, section 0.

Report tensile strength and modulus stress versus strain curve to failure, actual moulded thickness, actual and nominal thickness and Vf .

Validity

To be valid for design data, failure of the plain specimen must occur in the central region. If the damage extends into the end tab region the result will provide only a lower bound strength value.

303 METHOD OF TEST FOR THE NOTCHED TENSILE STRENGTH OF MULTIDIRECTIONAL FIBRE REINFORCED PLASTICS

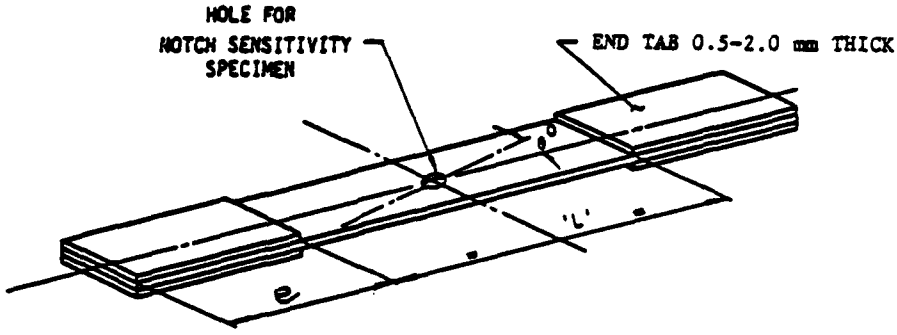


Fig 303.1

Description

This specimen is used to determine the notch sensitivity in tension of multidirectional laminates. The test may be used for either unidirectional tape or woven materials, provided the laminate is axially orthotropic to obviate induced bending. For basic strength and stiffness measurement a plain specimen is used (see Data Sheet 302). For comparative tests a specimen with a 5.0 mm diameter hole drilled at its centre has become standardised.

In the angled plies, no individual fibres should run under the tabs at both ends of the specimen. The free length is chosen so that a non-axial fibre can run across the full width of the specimen and be at least half its specimen width short of the end tabs at each end, *ie* minimum $L = w(1 + 1/\tan\theta)$.

For testing under dry ambient conditions soft aluminium alloy or GRP end tabs are suitable. For moist or hot conditions GRP end tabs are recommended. The tabs are attached using an adhesive suitable for the test environment.

The end tab adhesive shear strength will limit the level of load input and hence limit the amount of 0° fibres present in the laminate, *eg* typically for tests under dry ambient conditions a maximum thickness of 1.5 mm of 0° carbon fibres can be allowed.

Testing without end tabs is permissible provided that suitable end grips are used.

Nomenclature

e = length of end tabs

t = measured thickness of specimen

d = measured hole diameter

L = free length

θ = angle between fibres and longitudinal axis

P = load at failure.

Dimensions

Table 303.1

Recommended coupon widths

d mm	w mm
>4.0	30
>4.0 to 5.0	35
>5.0 to 6.0	40
>6.0 to 7.0	45 ±0.25 mm
>7.0 to 8.0	50
>8.0 to 9.0	55
>9.0 to 10.0	60
>10.0	6d

Holes may be filled with plain or C/S rivets or bolts.

e = 50 mm minimum

t = 1.0 to 4.0 mm depending on laminate configuration

L = not less than $w(1 + 1/\tan\theta)$, 2.5w or 100 mm whichever is the greatest.

For particular purposes other hole sizes, or filled holes with plain or C/S rivets or bolts may be used. A minimum w/d ratio of 6 is permissible.

For small holes (<4.0 mm) w may be reduced to between 20 mm and 30 mm provided that w is not less than 10t and 7d.

For small angles of $\theta(<15^{\circ})$ L may be reduced to $w/\tan\theta$.

Tolerances

Edges of specimen must be parallel to ±0.1 mm, specimen must be flat and end tab faces parallel and aligned to within ±0.05 mm.

Hole must be symmetrical about specimen width to ±0.1 mm.

Test requirements

If rivets are used they should be formed by the normal manufacturing process.

If bolts are used, a standard washer should be fitted under the nut, which should then be tightened to the normal manufacturing controlled torque level.

The specimen must be carefully aligned in test machine jaws to avoid induced specimen bending. Tensile load (or cross-head displacement) should be increased uniformly to cause failure within 30-90 seconds.

Calculations

Tensile strength for notched specimen (based on gross area) is given by:

$$f_{NT} = \frac{P}{wt} .$$

Report notched tensile strength, actual moulded thickness, and nominal thickness and V_f .

Validity

To be valid for design data, failure of the specimen must occur in the central region.

400 METHOD OF TEST FOR LONGITUDINAL COMPRESSION STRENGTH AND MODULUS OF UNIDIRECTIONAL FIBRE REINFORCED PLASTICS

Description

This Data Sheet defines the recommended method for measuring the longitudinal compression strength and modulus of a unidirectional laminate. After initial discussion by the group two methods were proposed but subsequent testing and investigation has led to the recommendation of a Celanese type specimen which is shown in Fig 400.1

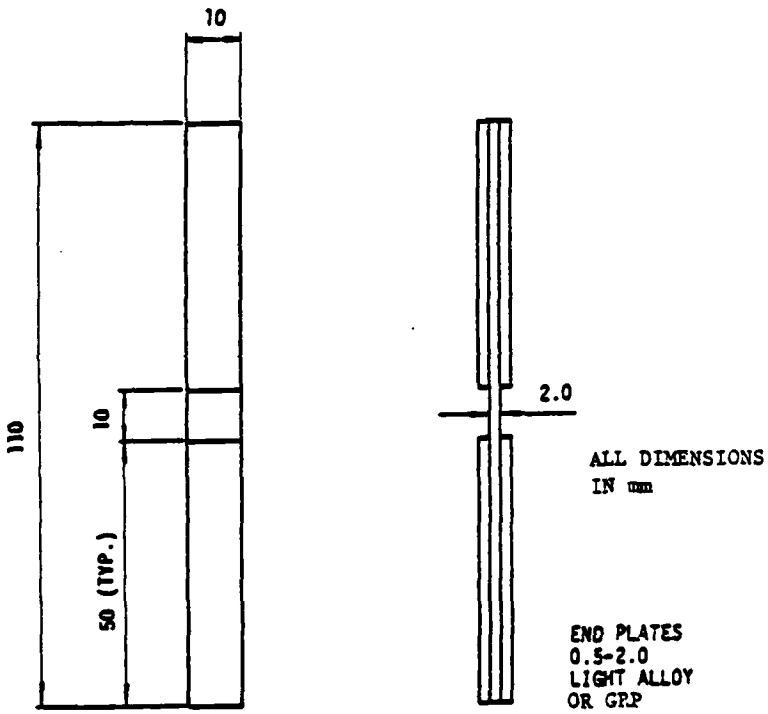


Fig 400.1 Unidirectional compression specimen

The specimen may be tested in a modified Celanese jig (based on ASTM D3419/75) for lateral restraint (*eg* see RAE TR 82047), or in a machine capable of maintaining accurate alignment and rotational restraint through the end fixtures.

The gauge length is 10 mm which is a compromise between the requirement to eliminate Euler buckling and the need to avoid end tab effects whilst providing adequate space for strain gauges.

The longitudinal modulus may be obtained from the strength test if the specimen is strain gauged. It is recommended that gauges be applied to both sides of the specimen in order to detect any Euler bending or loading eccentricity.

Nomenclature

t = measured thickness at specimen centre

w = measured specimen width

G.L. = gauge length

L = length of composite specimen

P = load at failure.

Dimensions

t = 2.00 mm or nearest mouldable thickness

w = 10.00 ± 0.25 mm

G.L. = 10.00 ± 0.25 mm

L = 110 mm.

Fibre alignment 0° ±30 seconds (relative to the specimen longitudinal axis).

Tolerances

Edges of specimens must be parallel to ±0.1 mm. Specimen must be flat and end tab faces parallel and aligned to within ±0.05 mm.

Test requirement

Compression load (or strain) should be increased uniformly to cause failure within 30-90 seconds.

Since some specimen bending may occur the average of the two surface strains must be taken as the axial strain.

Care must be taken during specimen manufacture because it is possible for end tab and glue line tolerances to accumulate and produce eccentric loading in the specimen.

012

Extreme care must be taken with specimen alignment in the machine to avoid off fibre-axis loading.

Calculations

Longitudinal compression strength is given by $F_{LC} = \frac{P}{wt}$ (1).

Accurately measured values of w and t must be used.

Since the stress versus strain graph is nonlinear, the compression modulus is defined as:

E_{1C} = secant modulus at -0/25% strain (1), (2).

Poisson's ratio = $\frac{\text{transverse strain}}{\text{axial strain}}$ at -0.25% strain (2).

NOTES: (1) The properties derived will be proportional to the fibre volume fraction of the laminate.

(2) For method of derivation, see Introduction section 0.

Report compression strength, modulus, stress versus strain curve to failure, fibre volume fraction (Vf) and width and actual and nominal thickness.

Validity

To be valid design strength data, failure must occur near the middle of the gauge length. Otherwise, the result will provide only a lower bound value.

401 METHOD OF TEST FOR LONGITUDINAL COMPRESSION STRENGTH AND MODULUS OF MULTI-DIRECTIONAL FIBRE REINFORCED PLASTICS

Description

This specimen is used to determine the compression strength and modulus of multidirectional laminates.

This Data Sheet does not refer to notched specimens which are dealt with by Data Sheet 402.

Unidirectional tape laminates or woven material specimens may be made in this configuration and tested provided the laminate is axially orthotropic to obviate bending or shear.

In the angle plies, no individual fibre should run under the end tabs of the specimen. The free length is chosen so that a non-axial fibre can run across the full width of the specimen and be at least half of its specimen width short of the end tabs, at each end, *ie*

$$L = W\left(1 + \frac{1}{\tan \theta}\right).$$

For testing under dry ambient conditions soft aluminium alloy or GRP end tabs are suitable. For moist or hot conditions GRP end tabs are recommended. The tabs are attached using an adhesive suitable for the test environment.

The end tab adhesive shear strength will limit the level of load and hence limit the amount of 0° fibres present in the laminate. Typically, for tests under ambient conditions, a maximum thickness of 1.5 mm of 0° carbon fibres can be allowed.

In exceptional circumstances testing without end tabs may be permissible provided that suitable end grips are used. The specimen is shown in Fig 401.1.

Nomenclature

- e = length of end tabs
- t = measured thickness of the specimen
- w = measured width of the specimen
- L = free length
- θ = angle between the fibres and longitudinal axis
- b = unsupported width between anti-buckling guide plates
- P = load at failure.

Dimensions

w = 9t minimum, typically 10t (absolute minimum = 20 mm)

e = 40 mm minimum

t = 2.00 to 4.00 mm depending on laminate configuration

b = see under Test requirements

L = not less than $w\left(1 + \frac{1}{\tan \theta}\right)$ or 100 mm whichever is the greatest.

Tolerances

The edge of the specimen must be parallel to within ±0.1 mm. Tolerance on thickness, ±0.05 mm.

The specimen must be flat and end tab faces parallel and aligned to within ±0.05 mm.

For small angles of $\theta(<15^{o})$ L may be reduced to $w/\tan \theta$.

Test requirements

The specimen must be carefully aligned in the test machine, and suitable anti-buckling guides employed. These guides should support as much of the specimen free length as possible, allowing for axial deformations, and should provide restraint against out-of-plane displacements at the ends to prevent local failure within the unsupported length. The distance 'b' between guide plates should ensure that the critical strain 'ε_{crit}' at which instability buckling may occur, as indicated in Fig 401.2, is always greater than the strain at which laminate compression failure is anticipated. The greater the margin of safety the less the possibility of premature instability induced failure. Whilst the guides are intended to support the specimen against out-of-plane deflection, care must be taken to avoid clamping friction and also any constraints to free Poisson's deformations across the width of the specimen. Forms of anti-buckling guides are shown in Figs 401.3 and 401.4.

Compressive load (or strain) shall be increased uniformly to cause failure within 30-90 seconds. For modulus determination the stress versus strain curves to failure must be recorded.

Calculations

Longitudinal compression strength is given by:

$$F_{LC} = \frac{P}{wt} .$$

Accurately measured values of w and t must be used.

Since the stress versus strain curve is nonlinear, compression modulus is defined as:

$$E_{IC} = \text{secant modulus at } -0.25\% \text{ strain (1)}$$

$$\text{Poisson's ratio} = \frac{\text{transverse strain}}{\text{axial strain}} \text{ at } -0.25\% \text{ strain (1)} \quad .$$

NOTE: (1) For method of derivation see Introduction section 0.

Report compression strength, compression modulus, stress versus strain curves, fibre volume fraction (Vf), nominal and actual moulded thickness.

Validity

To be valid for design data failure of the specimen must occur in the central region. If the damage extends into the end tab region the result will provide only a lower bound strength value.

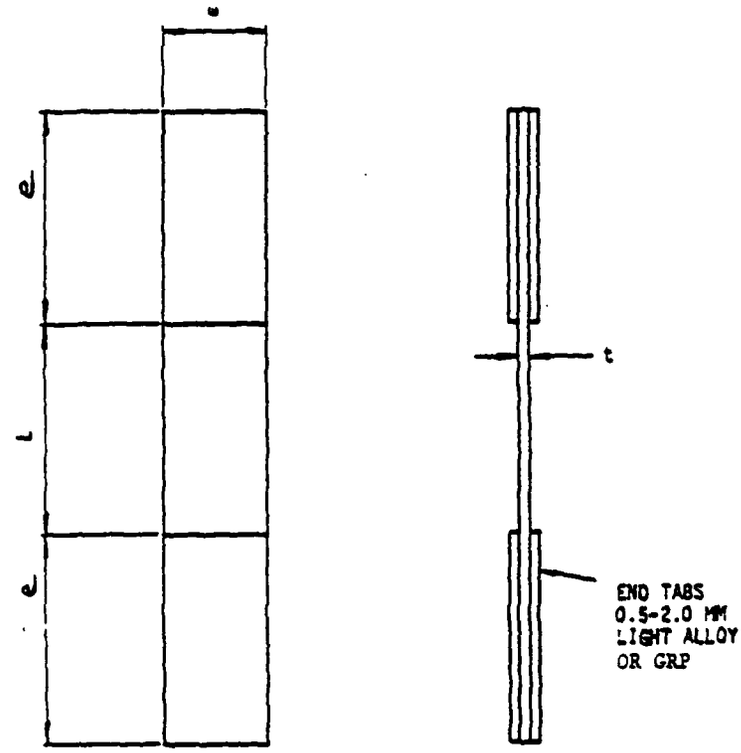


Fig 401.1 Multidirectional laminate: compressior. strength and modulus specimen

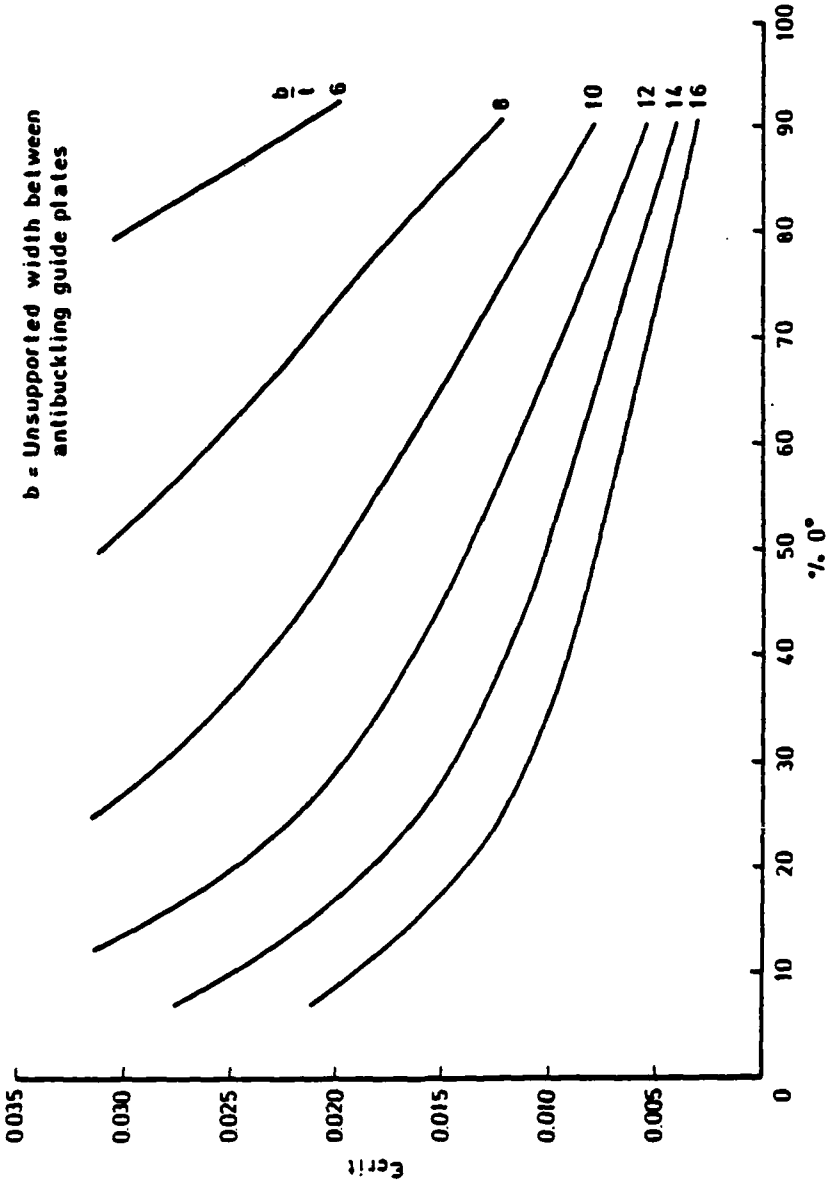


Fig 401.2 Instability of 0^{o}, $\pm 45^{o}$. 90^{o} family of laminates

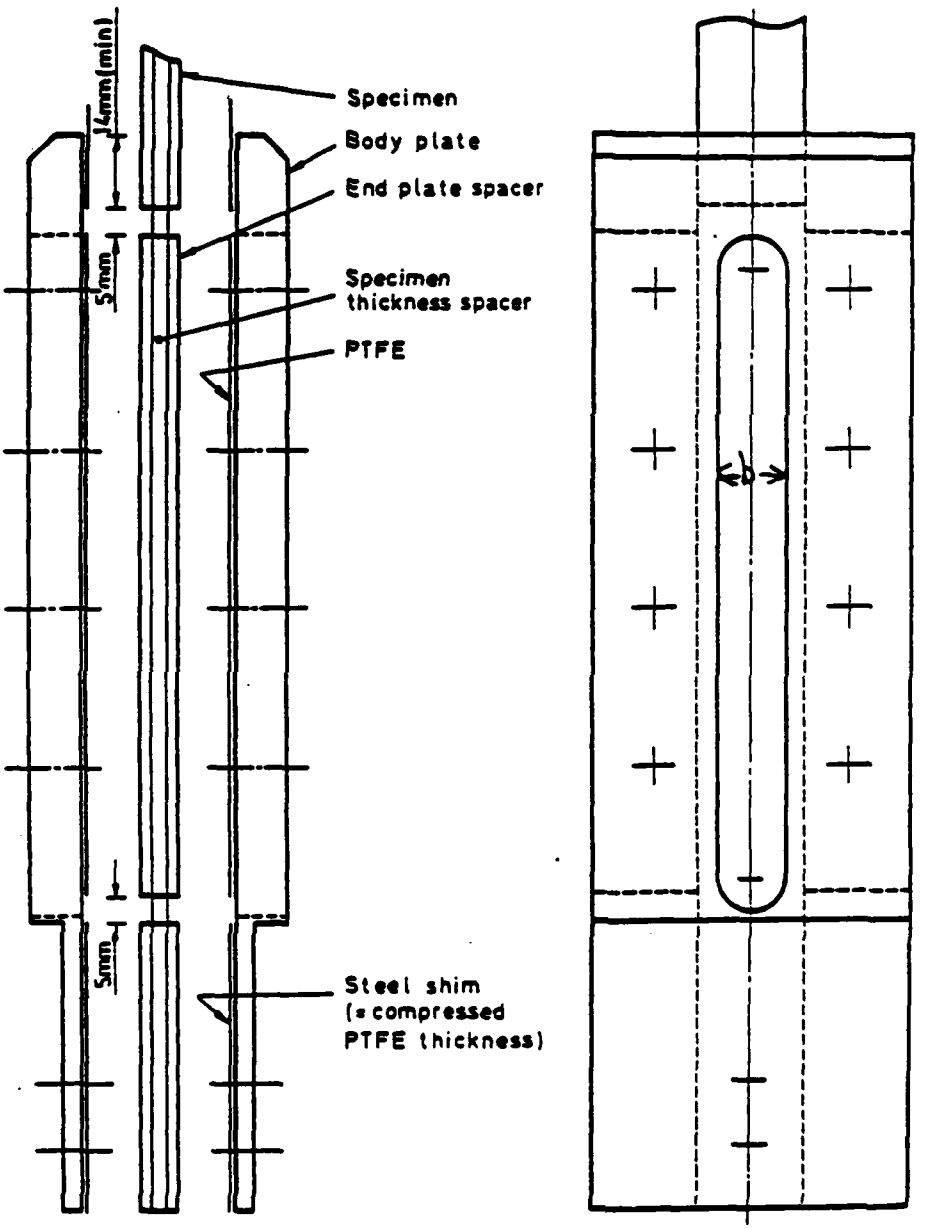


Fig 401.3 Anti-buckling guide

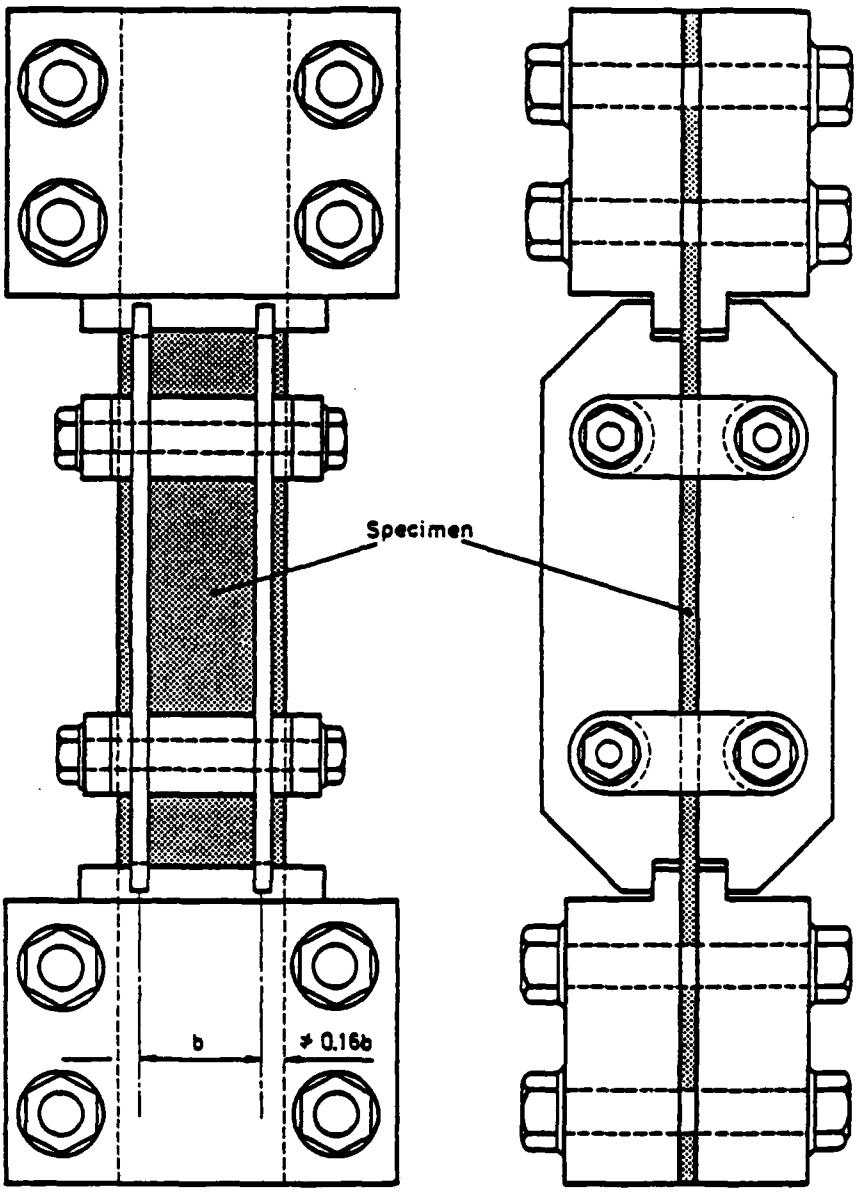


Fig 401.4 Anti-buckling guide

402 METHOD OF TEST FOR NOTCHED COMPRESSION STRENGTH OF MULTIDIRECTIONAL FIBRE REINFORCED PLASTICS

Description

This specimen is used to determine the compression strength of notched multidirectional laminates. In order to assess notch sensitivity, on a comparative basis, a specimen with a 5mm diameter hole drilled at its centre is used.

Unidirectional tape laminates or woven material specimens may be made and tested in this configuration provided that the laminate is axially orthotropic to obviate induced bending or shear.

In the angle plies, no individual fibres should run under the end tabs of the specimen. The free length is chosen so that a non-axial fibre can run across the full width of the specimen and be at least half specimen width short of the end tabs, at each end, *ie*

$$L = W\left(1 + \frac{1}{\tan \theta}\right) .$$

For testing under dry, ambient conditions, soft aluminium alloy or GRP end tabs are suitable. For moist or hot conditions GRP end tabs are recommended. The tabs are attached using an adhesive suitable for the test environment.

The end tab adhesive shear strength will limit the level or load input and hence limit the amount of 0° fibres present in the laminate, *eg* typically for tests under dry ambient conditions a maximum thickness of 1.5 mm of 0° carbon fibres can be allowed.

Testing without end tabs is permissible provided that suitable end grips are used.

The specimen is shown in Fig 402.1.

Nomenclature

- e = length of end tabs
- t = measured thickness of the specimen
- w = measured width of the specimen
- L = free length
- d = hole diameter
- θ = angle between fibres and longitudinal axis

P = load at failure

b = unsupported width between anti-buckling guides.

Dimensions

Table 402.1

Recommended coupon widths

d mm	w mm
<4.0	30
>4.0 to 5.0	35
>5.0 to 6.0	40
>6.0 to 7.0	45
>7.0 to 8.0	50
<8.0 to 9.0	55
>9.0 to 10.0	60
>10.0	6d

Holes may be filled with plain or C/S rivets or bolts.

e = 40 mm minimum

t = 2.0 to 3.0 mm depending on laminate configuration

L = not less than $W\left(1 + \frac{1}{\tan \theta}\right)$, 2.5w or 100 mm whichever is the greatest

b = see under Test requirement.

For particular purposes other hole sizes, or filled holes with plain or C/S rivets or bolts may be used. A minimum w/d ratio of 6 is permissible.

For small holes (>4.0 mm) w may be reduced to between 20 mm and 30 mm provided that w is not less than 10t and 7d.

For small angles $\theta(<15^{\circ})$ L may be reduced to w/tan θ .

Tolerances

The hole must be symmetrical about the specimen centre, ±0.1 mm.

The edge of the specimen must be parallel to within ±0.1 mm.

The specimen must be flat and end tab faces parallel and aligned to within ±0.05 mm.

Tolerance on width w ±0.25 mm.

Tolerance on thickness ±0.05 mm.

Test requirements

If rivets are used they should be formed by the normal manufacturing process.

If bolts are used, a standard washer should be fitted under the nut, which should then be tightened to the normal manufacturing controlled torque level.

The specimen must be carefully aligned in the test machine and a suitable anti-buckling guide employed (see Data Sheet 401).

NOTE: that compression load (or cross-head displacement) should be increased uniformly to cause failure within 30-90 seconds.

Calculations

Notched, longitudinal compression strength is given by:

$$F_{LC_n} = \frac{P}{wt} \text{ (based on gross area)} \quad .$$

Accurately measured values of w and t must be used.

Report compression strength, fibre volume fraction (Vf), nominal and actual moulded thickness.

Validity

To be valid for design data, failure of the specimen must occur in the central region. If the damage extends into the end tab region the result will provide only a lower bound strength value.

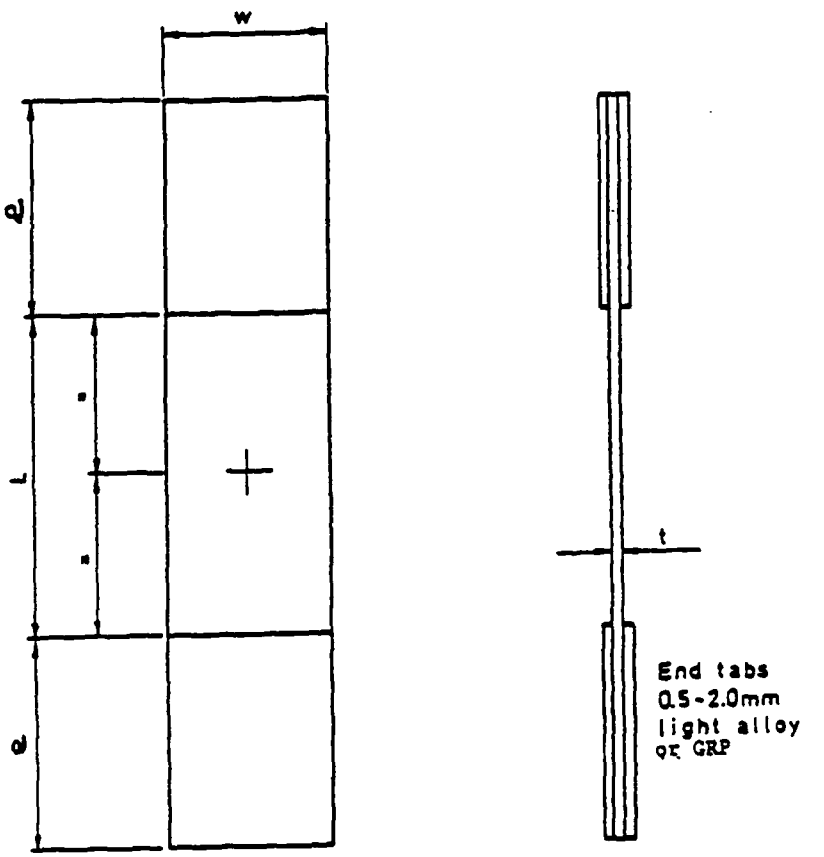


Fig 402.1 Multidirectional laminate: notched compression, strength specimen

403 METHOD OF TEST FOR RESIDUAL COMPRESSION STRENGTH AFTER IMPACT OF MULTI-DIRECTIONAL FIBRE REINFORCED PLASTICS

Description

This test method shall be used to determine the residual compressive strength of fibre reinforced plastics after impact damage.

The laminate thickness, ply orientation, specimen size and method of impact have been standardised for comparative purposes. For specific applications other laminate configurations and types of indentor may be used.

The tests shall be carried over in two stages:

(a) impacting the laminate over a range of energy levels and monitoring the type and size of damage produced (see Method 1001),

(b) testing damaged compression specimens cut from the test laminate to determine residual compressive strength.

Test laminate

The test laminate shall be quasi-isotropic (25%: 0°, 25%: 90°, 50%: $\pm 45^{\circ}$) and of the nearest moulded thickness to 3 mm. For unidirectional tapes of cured ply thickness 0.125 mm the lay-up shall be $(+45, -45, 0, 90)_{3S}$.

Impact tests

A suitable impact rig shall be used consisting of a drop-weight impacting onto the centre of an area of the test laminate securely clamped between two steel rings (see Fig 403.1). The clamp shall be designed to minimise the effect of uneven pressure distribution on the clamped area and shall be positioned on a solid non-energy absorbing surface.

The drop-weight shall consist of a mass with a steel ball or hemispherical indentor of radius in contact with the laminate d/2. The indentor shall be of 800 VPN minimum hardness and of surface finish less than 0.5 microns. The indentor shall be replaced at the first sign of damage or flattening.

The test laminate shall be impact damaged prior to being cut into compression specimens. The impacting of individual specimens shall not be allowed.

The drop-weight shall be released from the height (h) ensuring that it strikes the laminate at the centre of the clamped area. This may be achieved by the use of guides provided that frictional effects are negligible. The weight shall be caught on the rebound to prevent secondary damage to the laminate after initial impact.

The impacting of the laminate shall be carried out over a range of energies. The impact energy shall be adjusted by changing the mass of the drop-weight and keeping the height (h) constant. Each impact of the laminate shall be carried out in a new undamaged position. The centres of any damage positions shall not be closer than 100 mm to each other and to the edge of the laminate.

The extent and type of damage (*eg* delamination, splitting, fibre failure or penetration) shall be assessed visually and with the aid of ultrasonic inspection techniques (Method 1001). The impact energies at which damage first occurs (damage threshold) and at which damage is first visible to the naked eye on the laminate top surface, shall be recorded.

Areas of damage of width 40 mm or greater shall not be used for residual compressive strength testing. Any splitting of the lower outer ply of the laminate may be ignored provided the central delaminated area does not exceed 40 mm in width.

Compression tests

Compression specimens (see Fig 403.2) shall be cut from the laminate ensuring that the damaged area lies in the centre of the specimen.

For testing under dry ambient conditions soft aluminium alloy or GRP end tabs are suitable. For moist or hot conditions GRP end tabs are recommended. The tabs shall be attached to the specimens using an adhesive suitable for the test environment. Testing without end tabs is permissible provided that suitable end grips are used.

The specimen shall be carefully aligned in the test machine and a suitable anti-buckling guide employed (see Data Sheet 401). Compression load (or cross-head displacement) shall be increased uniformly to cause failure within 30-90 seconds.

Nomenclature

- e = length of end tabs
- h = drop height from indentor tip to the laminate surface in metres
- M = measured mass of drop-weight in kg
- U_I = impact energy in Joules
- d = indentor diameter

D_1 = clamping ring innder diameter

D_2 = clamping ring outer diameter

t = measured thickness of specimen

w = measured width of specimen

L = free length of specimen

b = unsupported width between anti-buckling guides

P = load of failure.

Dimensions

e = 40 mm minimum

h = 1 m

M = as required

d = 10 mm

D_1 = 100 mm

D_2 = 140 mm

t = 3 mm or nearest moulded thickness

w = 50 mm

L = 100 mm

b = 40 mm

Tolerances

The impact point shall coincide with the specimen centre line to within ±1 mm.

The edge of the specimen shall be parallel to within ±0.1 mm.

The specimen shall be flat and end tab faces parallel and aligned to within ±0.05 mm.

Tolerance on width w ±0.25 mm.

Tolerance on thickness t ±0.05 mm.

Calculations

Impact energy $U_I = M \times h \times 9.81$ Joules .

Residual compressive strength $F_{IC(r)} = \frac{P}{wt}$ (based on gross area).

Results shall be reported as a curve of residual compressive strength versus impact energy. Points shall be marked on the curve indicating the energy at which damage first occurs (damage threshold) and the energy at which damage is first visible to the naked eye on the top surface of the laminate.

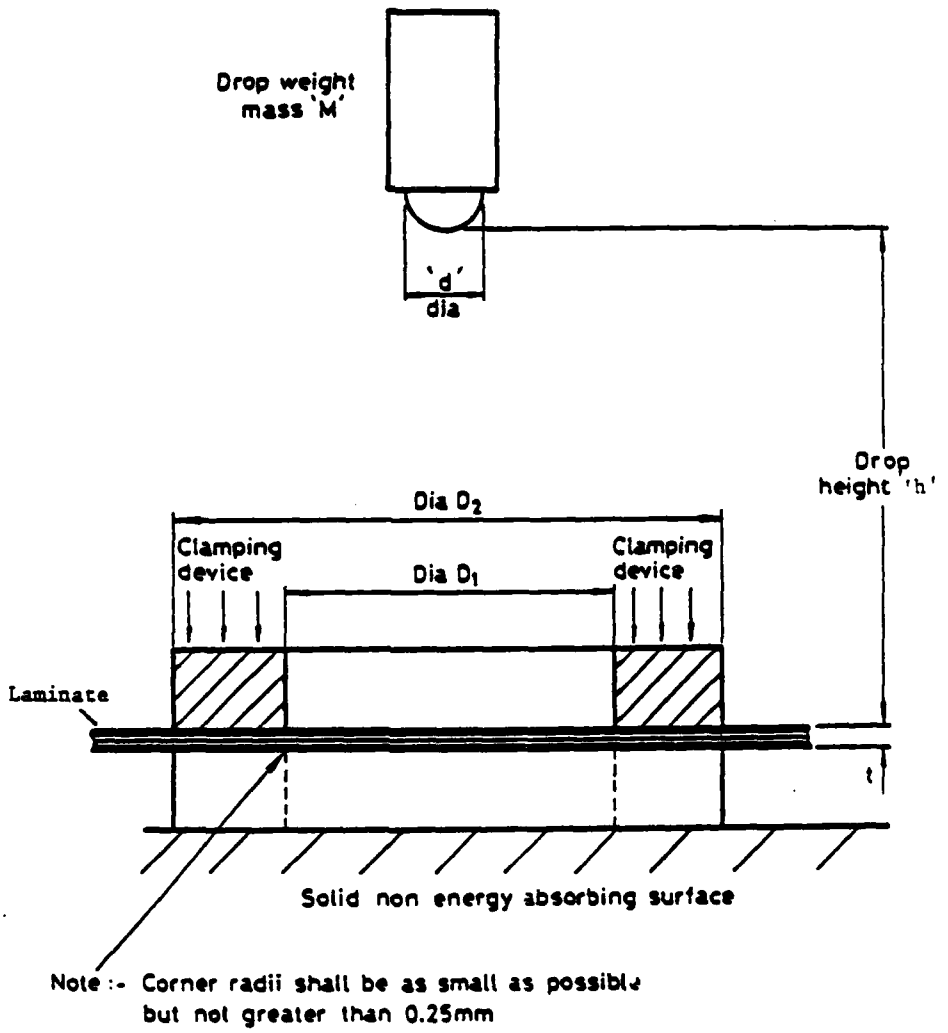


Fig 403.1 Schematic diagram of the laminate clamp and dropweight

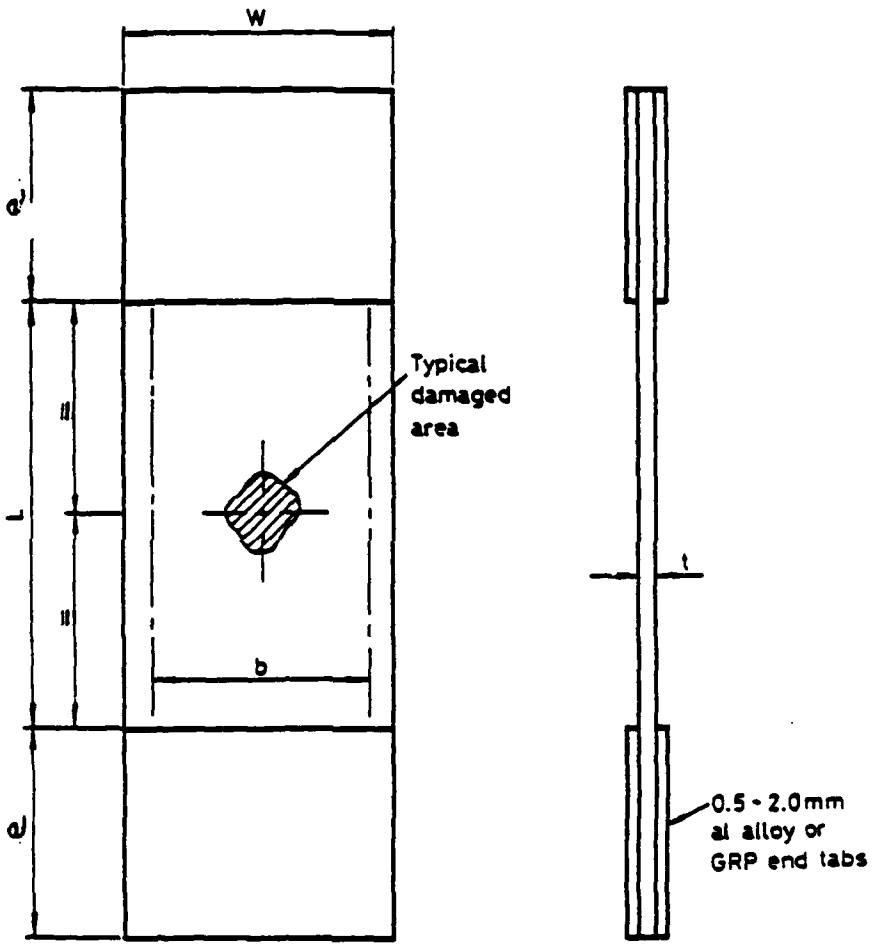


Fig 403.2 Impact damaged compression specimen

500 METHODS FOR THE PREPARATION OF TEST SPECIMENS FOR THE MEASUREMENT OF FATIGUE PROPERTIES OF FIBRE REINFORCED PLASTICS

Description

The preceding methods deal primarily with the static properties of composite materials. However, similar test specimens are suitable for measuring fatigue performance. This method describes the applicability of the various specimens for fatigue testing.

Interlaminar shear test

In general, the roller sizes and specimen dimensions outlined in Method 100 are satisfactory for fatigue testing, but opposed rollers are usually required, particularly for reversed cycling. When using pairs of rollers particular care must be taken to prevent an excessive frictional moment developing, due to the clamping effect of the opposed rollers.

Detection of failure has been found difficult, particularly for GRP, since there is often very little change in specimen deflection at failure. The use of acoustic emission equipment as a means of failure detection has proved useful in this respect, the onset of failure coinciding with a marked increase in the rate of acoustic output.

Flexural testing

The specimen design and test configuration defined in Method 200 may be used, but as for the ILSS specimen opposed rollers are usually necessary in fatigue. In designing a jig for flexural fatigue testing care should be taken to ensure that the rollers can pivot to obviate any clamping moment due to angular deflection at the specimen ends. Care must also be taken to avoid fretting under the loading rollers.

Axial testing

The plain specimens defined in Methods 300-303 and Methods 400-402, with a total length of 250 mm, have proved satisfactory for the tension/compression fatigue testing of fibre composites. The presence of waisting in the width and thickness may be slightly beneficial for GRP specimens with predominantly axial fibre and tested under dry ambient conditions. In all other cases, waisting is not recommended because of the possibility of premature failure due to shear cracks initiated at the shoulder of the waists.

If a compressive excursion is included in the fatigue cycle, it is necessary to provide supports to prevent buckling. The guide depicted in Fig 401.3 has

proved satisfactory. Studies to optimise the design of anti-buckling supports, however, are still proceeding but some general guidelines that should be adhered to can be given:

(a) The free, unsupported, area of the specimen should be a maximum, consistent with the requirement of preventing buckling, so as not to restrict any anticipated failure modes.

(b) Friction between the supports and the specimen must be minimal (PTFE tape on the contact surfaces is recommended).

(c) To ensure the anti-buckling supports are not loaded, sufficient gaps between the ends of the supports and the grips of the test machine must be available to accommodate the maximum specimen strain.

As a less preferable alternative, short stable specimens may be used for compressive and reversed cyclic fatigue tests. The disadvantage is that the stress distribution in the short free length may be affected by the restraint at the grips. Reducing the specimen width to allow for this renders the edge stresses more critical. Typically such specimens will be about 10 mm wide with a 10 mm free length. The minimum thickness is about 1.5 mm.

Induced heating

A problem associated with the fatigue testing of composites is the effect of mechanical heating. This is most critical in matrix dependent behaviour (*eg* ±45 degree lay-ups and ILSS specimens). The specimen and test rig should be arranged to disperse this heat where possible. Aluminium alloy end tabs attached with an aluminium filled adhesive will assist. The rate of cycling should be restricted, or if necessary forced cooling provided, to maintain the required uniform specimen temperature. As a guide, matrix dependent coupons should be cycled at about 5 Hz or less but for other coupons 10 Hz or more is usually acceptable.

600 METHOD OF TEST FOR INTERLAMINAR FRACTURE TOUGHNESS OF FIBRE REINFORCED PLASTICS

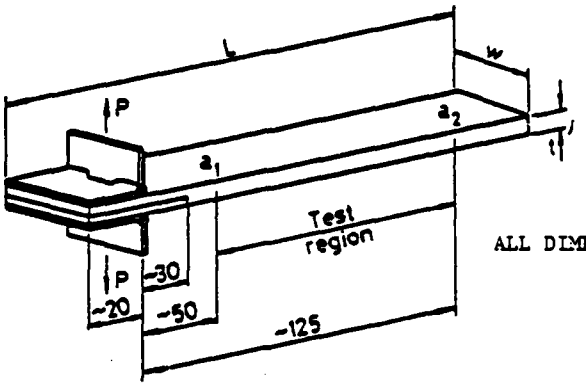


Fig 600.1 Interlaminar fracture toughness test using double cantilever beam specimen

Description

This specimen is used to obtain a value for fracture toughness of a matrix resin - fibre combination and is of principal benefit in the qualitative comparison of different resins or fibre systems. The method may also be useful in the study of delamination growth models. The specimen consists of a rectangular double cantilever beam, one end of which is prised apart by means of bonded hinges. The energy required to delaminate an area between the two central plies of the specimen is measured and used to calculate the energy release rate G_{1c}.

The start of a crack is helped by the insertion of a layer of thin teflon or similar separator during laminate lay-up and the metallic hinges are bonded to the specimens using a suitable room temperature curing adhesive.

Some means of mechanical fastening through the specimen half thickness may be required if the material toughness is too great for the adhesive available.

The specimens may be made from unidirectional or woven reinforcement. They must contain an even number of plies and in the case of woven material have a lay-up symmetrical about the thickness centre line.

If inter-fibre interference is a problem, then the unidirectional specimen may be assembled with alternate plies at $\pm 5^{\circ}$ to the specimen longitudinal direction.

Nomenclature

t = measured thickness of test section (mm)

W = average width in test region (mm)

L = specimen length (mm)

P = load recorded during test procedure in N

a_1 = average distance between the hinge pin and initial crack tip (mm)

a_2 = average distance between the hinge pin and final crack tip (mm)

A = integrated area under the load/deflection curve less the area represented by the energy remaining in the specimen (N.mm).

Dimensions

The thickness t should be selected such that it is approximately equal to $21.25 \times (G/E_f)^{1/3}$ where G is the estimated energy release rate and E_f the composite flexural modulus in the specimen L direction. Where an estimate of G is not possible use, initially, a value of t = 3 mm.

$$w = 38 \pm 0.5 \text{ mm}$$
$$L = 225 \pm 5 \text{ mm} .$$

Test requirements

Mount the specimen in the test machine by gripping the upstanding legs of the hinges. Increase the load at 5 mm per minute until the crack length reaches approximately 50 mm. Stop the machine, unload the specimen and with the aid of a stereo microscope, mark the crack tip on both edges of the specimen (note removal of the specimen may be necessary). The average of the lengths from both sides is designated as a_1 .

Continue the test, replacing the specimen in the test machine if removed, and record load versus displacement. Extend the crack length to approximately 125 mm. Stop the machine, remove the specimen and, as above, identify the average final crack tip length, a_2 . Identify also these points on the load/displacement chart (see Fig 600.1).

Calculation

Fracture toughness (energy release rate).

G_{ic} is given by

$$G_{ic} = \frac{A}{w(a_2 - a_1)} 10^3 \quad Jm^{-2}$$

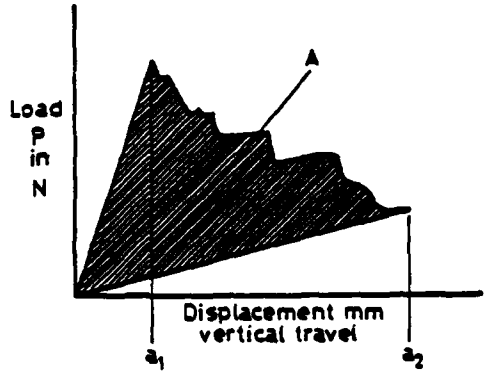


Fig 600.1 Report fracture toughness, fibre volume fraction

Validity

For valid comparisons, resin should be tested with identical fibres or fabrics at identical fibre volume fractions.

Similarly different fibres or fabrics should be compared with the same matrix resin.

700 METHOD OF TEST FOR BEARING PROPERTIES OF MULTIDIRECTIONAL FIBRE REINFORCED PLASTICS

Description

This specimen is used to determine the bearing strength of multidirectional laminates and to measure the bearing stress versus hole deformation. It may be made from undirectional tape, woven material or mixtures thereof, provided that the laminate is axially orthotropic to obviate induced bending.

The specimen may be used for tension or compression tests: with many laminates different strength values will be obtained.

The specimen is shown in Fig 700.1.

Nomenclature

- t = thickness of specimen
- d = hole diameter
- w = width of specimen
- L = length of specimen
- e = distance from centre of hole to the free end of the specimen
- P = applied load (tension or compression)
- P_F = applied load at failure
- Δ = measured deformation over gauge length
- σ_{bult} = bearing stress at failure.

Dimensions

Values for w and e for various hole diameters are given in the following table.

d mm	w mm	e mm
<4	30	24
>4-5	35	30
>5-6	40	36
>6-7	45	42
>7-8	50	48
>8-9	55	54
>9-10	60	60
>10	6d	6d

t = between d/3 and d
L = to suit test fixture.

Tolerances

The hole must be on the centre line of the specimen to within ±0.1 mm. The hole tolerances should conform to BS 4500 H10 (metric) or BS 1916 H10 (imperial). The specimen must be flat to within ±0.05 mm.

Permitted deviations

For small holes (4 mm) w may be reduced to between 20 mm and 30 mm provided that w is not less than 10t and 7d.

For specific purposes values of t outside the recommended range may be used. However with lower values the average bearing stress at failure may be affected by plate instability, whilst with higher values the average bearing stress at failure may be affected by bolt bending.

Test requirements

The bolt through the specimen should be torque tightened to provide a level of lateral constraint appropriate to the practical application to which the test relates. A low constraint will permit a local 'brooming' failure, giving a brushlike appearance around the loaded half of the hole. With higher levels of constraint 'brooming' is prevented giving an increase in the ultimate bearing stress.

For tension tests, a suitable test fixutre is illustrated in Fig 700.2.

For compression tests the same fixture may be used with compression jaws in the test machine, or alternatively between platens with accurately parallel end blocks.

The specimen must be carefully aligned in the test fixture and machine to ensure axial loading.

When load versus hole deformation measurements are required a suitable extensometer must be fitted to record the movement between the free end of the specimen and bearing bolt in the test fixture.

The load should be increased uniformly to cause failure within 30-90 seconds.

Calculations

The ultimate bearing stress is calculated from the formula:

$$\sigma_{bolt} = \frac{P}{dt} .$$

Bearing stress versus hole deformation data should be given in the form of bearing stress versus % elongation of the hole, *ie*

$$\frac{P}{dt} \text{ versus } \frac{\Delta}{d} 100 \quad .$$

Accurately measured values of d and t must be used.

Report bearing stress at failure, bearing stress versus % elongation, nominal fibre volume fraction, nominal and actual moulded thickness, bolt diameter, bolt material, level of torque tightening, and any other significant features, *eg* if transverse restraints are used.

Validity

To be valid bearing data, all damage must lie within the boundaries of the specimen. Shear out or total failure of the specimen do not constitute valid results.

Note that in specimens with large percentages of 0° fibres, splitting parallel with the fibres can lead to premature shear out failure. This can be prevented by applying transverse restraint to the specimen by means of edge supports. In so doing however the validity of the test in relation to the application it represents must be considered.

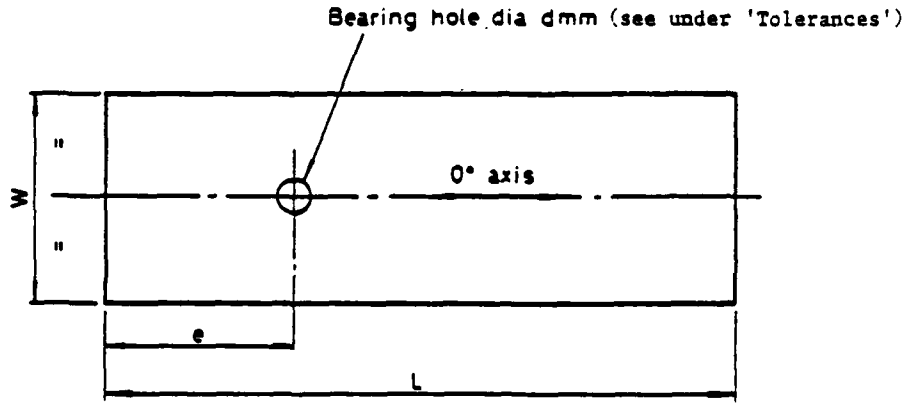


Fig 700.1 Test specimen

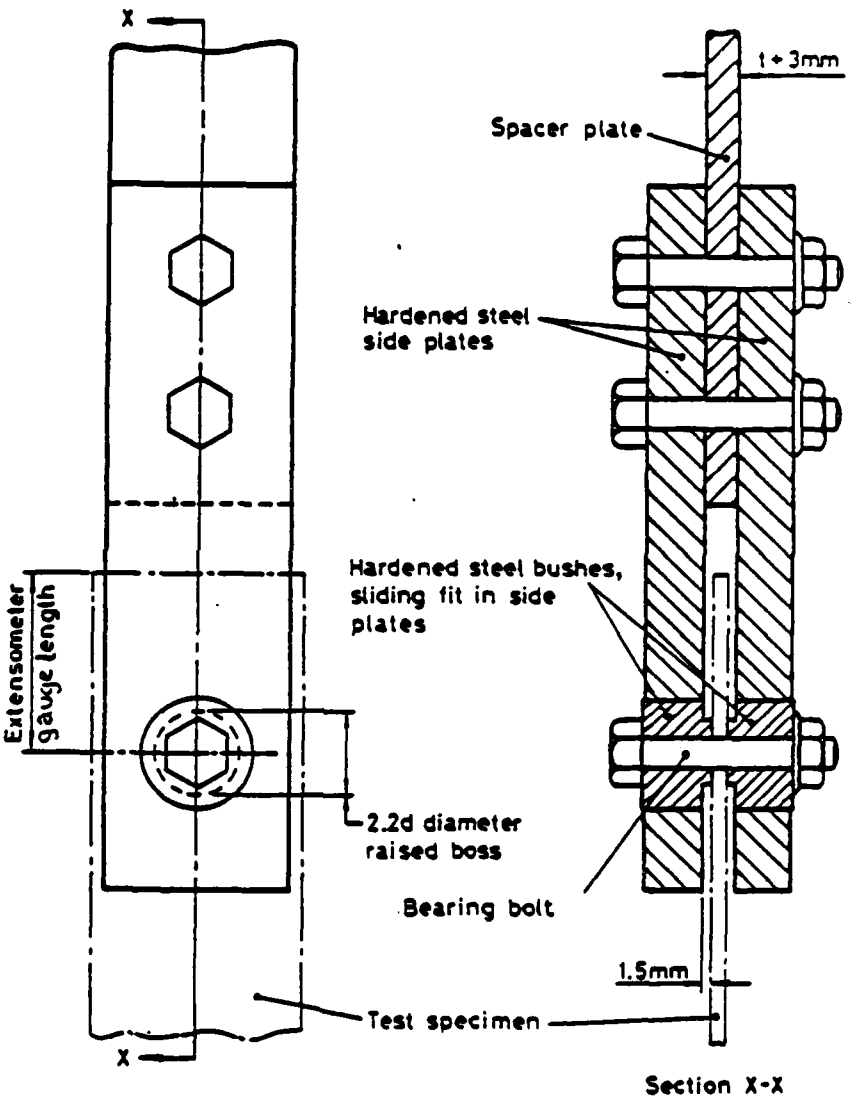


Fig 700.2 Test fixture

800 METHOD OF TEST FOR THE DENSITY OF FIBRE REINFORCED PLASTICS

Description

In principle, there should be no significant difference between the techniques required for the measurement of the density of unreinforced plastics and of fibre reinforced composites. Thus any method based on the displacement technique, such as BS 2782 Part 6 method 620A, should be suitable. However, values of composite density are usually required for volume fraction and void content calculations, and for this an accuracy of at least ±0.2% is desirable. This accuracy can only be attained in the displacement method described by thorough care and attention to detail. Measurements made by density gradient column are unlikely to be sufficiently accurate.

Method

The displacement method, as described in BS 2782 Part 6 method 620A, involves the weighing of a small sample of composite, typically about 1 g or greater. The sample should be thoroughly dried, as described in Method 901 describing the measurement of diffusivity, or used immediately after manufacture. Weighings both in air and immersed in a fluid, of lower density than the sample, should be taken to an accuracy of 0.1 mg. Care should be taken to ensure no air bubbles adhere to the test sample. The use of a wetting agent may assist if this proves to be a problem, but the effect of this on the density of the immersion fluid must be taken into account. The density of the test sample is calculated using the following equation:

$$\rho t = a\rho x/(a - b)$$

where a is the mass, in grams, of the test sample in air and b the apparent mass of the test sample immersed in the fluid. ρx is the density of the fluid in g/cc, which if water is used as the immersion fluid is 0.9975 at 23°C. The fluid temperature should be maintained to better than ±2°C. Corrections will need to be applied if the immersion liquid is not maintained precisely at this temperature.

Test report

The test report should include the following particulars:-

(a) Complete identification of the material tested, including fibre and resin type, manufacturer, lay-up and stacking sequence, plus any previous history (including drying procedure).

(b) Sample dimensions.

(c) Reference to the method employed (*eg* BS 2782, Method 620A).

(d) Density as calculated from the above equation, together with the test temperature and details of the immersion fluid.

801 METHOD OF TEST FOR THE DETERMINATION OF THE COEFFICIENT OF LINEAR THERMAL EXPANSION OF FIBRE REINFORCED PLASTICS

Description

This Data Sheet defines the recommended method for measuring the coefficient of linear thermal expansion. The method is based upon ASTM D696-79 and E228-79.

The method may be applied to unidirectional, multidirectional and woven laminates. However, because of the directional influence of the reinforcement, their interaction with each other and the matrix, it is necessary to ensure sufficient specimens are evaluated to accurately determine the property in the specified direction of the material or laminate.

The coefficient of linear thermal expansion is determined by use of a dilatometer having the critical expansion components constructed from vitreous silica. The principle is that the specimen is placed at the bottom of the outer tube with the inner tube resting on it. Variation in the length of the specimens, due to temperature changes, are measured by the differences occurring between the inner and outer tubes (Fig 801.1). The temperature changes are brought about by immersing the dilatometer in a liquid bath accurately controlled at the desired temperature.

At the test conditions the materials shall have negligible creep or elastic strain rate.

The expansion of the specimen is influenced by moisture content; to eliminate this phenomenon the specimen must be pre-dried to a constant weight, see Method 901. Expansion is also affected by stress relaxation, transitional and phase changes; these effects can be reduced by pre-conditioning of the specimens and selection of a suitable range of test temperatures.

The ends of the specimens shall be flat and perpendicular to the length axis, and may be protected against indentation by thin steel shims (0.012 to 0.020 inch thick) bonded with adhesive suitable for the test environment. Details of the specimen geometry are given in Fig 801.2.

Nomenclature

Lo = length of specimen at room temperature

L1 = length of specimen at T1

L2 = length of specimen at T2

T1 = lower temperature of assessment

T2 = higher temperature of assessment

α = coefficient of linear thermal expansion per degree Celsius

ΔL = change in length of specimen due to heating or cooling

ΔT = temperature difference over which change in length was measured.

Test requirements

Specimens must be carefully aligned in the dilatometer to prevent friction. The temperature stability of the specimens is indicated by a nil movement of the measuring device over a period of 5-10 minutes at the test temperature. The top of the specimen must be at least 50 mm below the level of the bath.

The open end of the dilatometer shall be at least 50 mm above the liquid level.

Calculations

The coefficient of linear thermal expansion is given by

$$\frac{\Delta L}{L_o \Delta T} .$$

Report

The form and dimensions of the test specimens, the type of apparatus, temperatures between which α was determined and average α per degree Celsius should be reported.

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Report

The form and dimensions of the test specimens, the type of apparatus, temperatures between which α was determined and average α per degree Celsius should be reported.

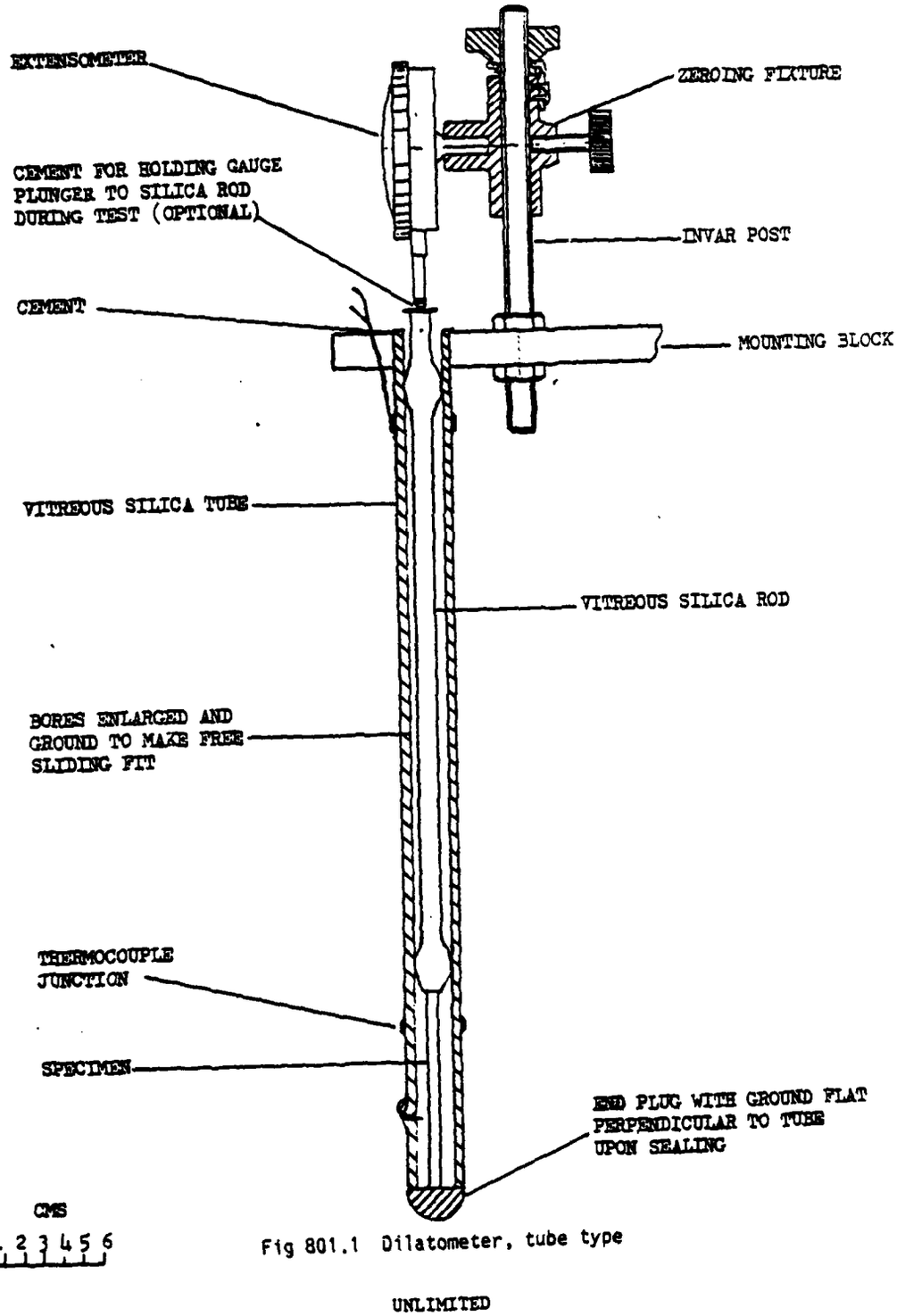

Fig 801.1 Dilatometer, tube type

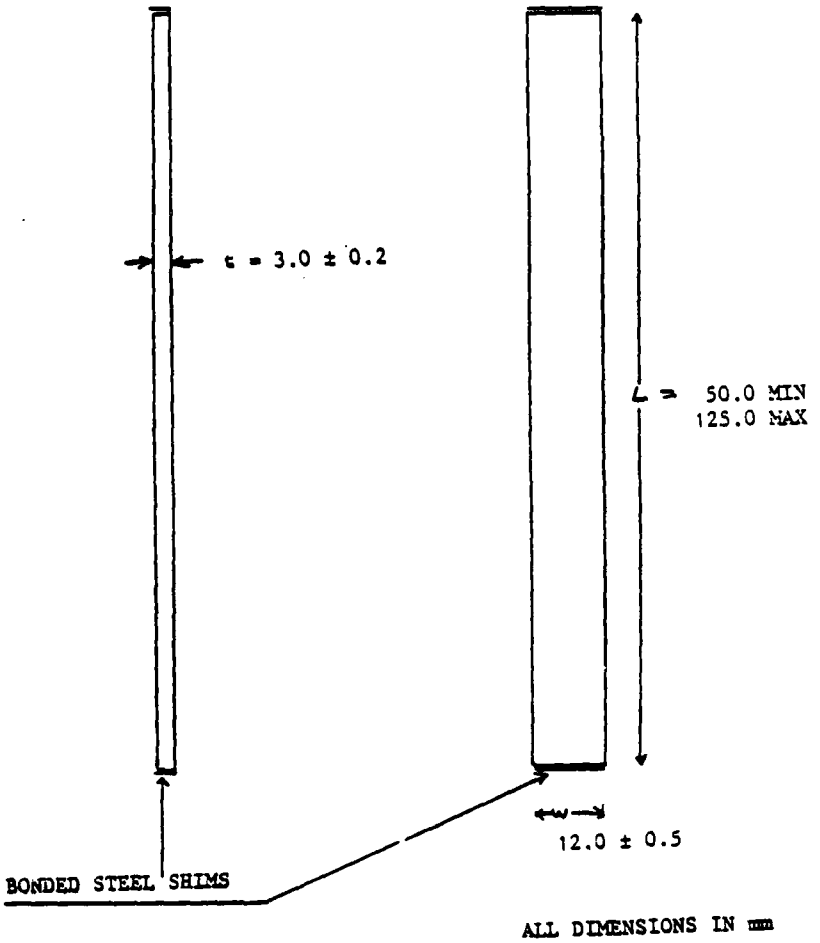

Fig 801.2 Specimen configuration

802 METHOD OF TEST FOR THE OUTGASSING OF FIBRE REINFORCED PLASTICS

1 Scope

The test method covers a screening technique to determine the volatile content of materials exposed to a vacuum environment at elevated temperature. Three parameters are measured: total mass loss (TML), collected volatile condensable materials (CVCM), and the recovered mass loss (RML). The method describes the test apparatus and related operating procedures for evaluating the mass loss of materials being subjected to 125°C at less than 7×10^{-3} Pa (5×10^{-5} torr) for 24 hours. The overall mass loss can be classified into condensables and non-condensables. The former are characterized herein as being capable of condensing on a collector at a temperature of 25°C.

This procedure is intended for the testing of composite materials for use in a space environment. The method is primarily a screening technique for materials and is not necessarily valid for computing actual contamination on a system or component because of differences in configuration, temperatures, and material processing. The criteria used for the acceptance and rejection of materials shall be determined by the user and based upon specific component and system requirements.

2 Definitions

Collected volatile condensable material, CVCM, is the quantity of outgassed matter from a test specimen that condenses on a collector maintained at a specific constant temperature for a specified time. CVCM is expressed as a percentage of the initial specimen mass and is calculated from the condensate mass determined from the difference in mass of the collector plate before and after the test.

Total mass loss, TML, is total mass of material outgassed from a specimen that is maintained at a specified constant temperature and operating pressure for a specified time. TML is calculated from the mass of the specimen as measured after pre-conditioning and immediately after completion of the test and is expressed as a percentage of the initial specimen mass.

Recovered mass loss, RML, is the mass loss measured after the specimen has been dried in accordance with procedures laid down in Method 901 of this document and allowed to pre-condition at 20°C and 65% relative humidity for 24 hours following the testing. This RML is determined after TML has been measured. RML is calculated from the mass of specimen as measured after pre-conditioning of the

test and after post-conditioning following testing expressed as a percentage of the initial specimen mass.

3 Summary of method

The dried test specimen is pre-conditioned to 20°C and 65% relative humidity for 24 hours in a preformed, degreased container (boat) that has been weighed. After this exposure, the boat and specimen are weighed and put in one of the specimen compartments in a copper heating-ring that is part of the test apparatus. The copper heating-ring can accommodate a number of specimens for simultaneous testing. The vacuum chamber in which the heating-ring and other parts of the test apparatus are placed is then sealed and evacuated to a vacuum of at least 7×10^{-3} Pa (5×10^{-5} torr). The heating-ring is used to raise the specimen compartment temperature to 125°C. This causes vapour from the heated specimen to stream from the hole in the specimen compartment. The vapour passes into a collector chamber in which some vapour condenses on a previously-weighed and independently temperature-controlled, chromium-plated collector plate that is maintained at 25°C. Each specimen compartment has a corresponding collector chamber that is isolated from the others by a compartmented separator plate to prevent cross-contamination. After 24 hours, the test apparatus is cooled and the vacuum chamber is repressurized with a dry, inert gas. The specimen and the collector plates are weighed. From these results and the specimen mass determined prior to the vacuum exposure, the percentage TML and percentage CVCM are obtained. Normally, the reported values are an average of the percentages obtained from three samples of the same material.

NOTE: It is also possible to conduct infra-red and other analytical tests on the condensates in conjunction with mass-loss tests. Infra-red transparent flats may be used for infra-red analysis. These flats are nominally 25 mm (1 inch) in diameter by 3.2 mm (0.125 inch) thick, and are supported edgewise in a metal holder that fits into the collector plate receptable. On completion of the test, the flats are placed into an infra-red salt flat holder for examination by an infra-red spectrophotometer. As an alternative method, the condensate may be dissolved from the metallic collector, the solvent evaporated, and the residue deposited on a salt flat for infra-red tests. Infra-red transparent flats shall not be used for CVCM determinations.

After the specimen has been weighed to determine the TML, the RML can be determined, as follows. Post-condition the specimen for 24 hours at 20°C

and 65% relative humidity to permit sorption of water vapour. The specimen mass after this exposure is determined. From these results and the specimen mass prior to vacuum exposure, the percentage RML is obtained.

Three empty specimen chambers and collector plates in the heater-ring shall be used as controls to ensure that adequate cleaning procedures have been followed after each test.

A typical test apparatus can have as many as 24 specimen chambers with 24 associated collector plates so that a number of specimens of different types can be tested each time the foregoing operations are conducted. Three specimen compartments shall serve as controls and three can be used for each type of material being tested. The equipment shall be calibrated at least once a year by using previously tested materials as test specimens or by round-robin tests between facilities.

The equipment shall be considered acceptable if the results meet the following criteria:

CVCM/TML/RML > 0.2% within ±20% (relative) of 'standard' value

CVCM/TML/RML < 0.2% within ±0.05 (absolute) of 'standard' value .

The apparatus may be oriented in any direction as long as the configuration shown in Fig 802.1 is maintained and bulk material does not fall from the sample holder nor obstruct the gas exit hole. The dimensions for critical components given in Fig 802.1 and Table 802.1 are provided so that apparatus constructed for the purpose of this test may provide uniform and comparable results.

4 Significance and use

This test method evaluates, under carefully controlled conditions, the changes in the mass of a test specimen on exposure under vacuum to a temperature of 125°C and the mass of those products that leave the specimen and condense on a collector at a temperature of 25°C. Comparisons of material outgassing properties are valid at 125°C only. Samples tested at other temperatures may be compared only with other materials which were tested at that same temperature.

The measurements of the collected volatile condensable material are also comparable and valid only for similar collector geometry and surfaces at 25°C. The simulation of the vacuum of space in this test method does not require that the pressure be as low as that encountered in interplanetary flight (for example,

10^{-12} Pa (10^{-14} torr)). It is sufficient that the pressure be low enough that the mean free path of gas molecules be long in comparison to chamber dimensions.

This method of screening materials is considered a conservative one. It is possible that a few materials will have acceptable properties at the intended use temperature but will be eliminated because their properties are not satisfactory at the test temperature of 125°C. Also, materials that condense only below 25°C are not detected. The user may designate additional tests to qualify materials for a specific application.

5 Apparatus

The apparatus used in the determination of TML and CVCM typically contains two resistance-heated copper rings. The lower ring contains 24 specimen chambers, the upper ring 24 holes. The open section of the upper ring allows vapours from the specimen to pass through a hole into a collector chamber where it impinges on a removable chromium-plated collector plate maintained at 25°C throughout the test, (see Figs 801.1 and 801.2). Variations in test apparatus configurations are acceptable if critical dimensions are maintained as prescribed in Table 801.1.

The operation of the vacuum chamber system and any device for raising the vacuum bell can be automatically controlled. Power to the heating element mounted in the copper rings is generally controlled by variable transformers through temperature controllers. Recorders with an electronic icepoint reference junction feedback may be used to monitor the heater ring temperatures. A heat exchanger using a suitable fluid may be used to maintain the collector plate at 25°C during the test.

It is recommended that the vacuum chamber system include automatic controls to prevent damage in the event of power failure when in unattended operation. Care must be taken to prevent backstreaming of oil from vacuum or diffusion pumps into the vacuum chamber.

6 Test specimens

Test specimens shall be protected from contamination by packaging them in clean new polyethylene bags after preparation and for transportation to the test facility.

Test specimens shall be supplied preferably as 1.5 to 2.0mm thick laminates. Thicker specimens may be submitted but must be cut dry, into 1.5 to 2.0mm thick sheets. The sheets must be cut dry to the final size of material for testing which shall be cubes of 1.5 to 2.0 mm side. Minimum specimen masses in the

order of 200 mg are required. If smaller quantities are utilized, the accuracy of the measurements may be impaired.

It is absolutely essential that specimen materials not be contaminated at any step in the specimen fabrication process. Most importantly, specimen material shall not be handled with the bare hands as oils from human skin are volatile and condensable and thus will cause false TML and CVCM results.

To control contamination, suitable gloves or finger cots shall be worn during all specimen preparation steps. All materials will be tested in the 'as-received' condition, unless a cleaning schedule is advised.

Only the use of cleaning solvents that are known to be non-reactive with the specimen material and that leave no residue are permitted.

7 Procedure

7.1 Weigh a prepared nickel boat (M_c).

7.2 Add the test specimen (100-300 mg) to the boat and pre-condition the sample at 65% relative humidity and 20°C for 24 hours.

7.3 Weigh the pre-conditioned specimen and boat (M_o),

$$\text{sample weight} = (M_o - M_c) \quad .$$

7.4 Weigh a prepared collector and mount it into its cooling-plate receptacle (M_p).

7.5 Place the specimen and boat into a specimen compartment of the heating-ring in the microvolatile condensable system.

NOTE: Prior to the operation noted in procedure 7.5, the copper compartment ring, separator, and cooling plate shall be clean, in position, and awaiting the specimen boats and collector-plates.

7.6 Close the vacuum system and evacuate it to 7×10^{-3} Pa (5×10^{-5} torr) or less within 1 hour.

7.7 Control of the collector-plate temperature at 25°C shall be achieved within the first hour of pump-down.

7.8 When a pressure of 7×10^{-3} Pa (5×10^{-5} torr) is reached, turn on the heater-ring and adjust the variable transformers to raise the heater-ring temperature to 125°C within 60 minutes.

7.9 Maintain the collector-plate temperature at 25°C.

7.10 Maintain the heater-ring temperature at 125°C for 24 hours, then close the high vacuum valve to the pumping system and turn off the heater-ring.

7.11 Open the vent valve and backfill with white spot nitrogen regulated within a gauge pressure range from 10-30 kPa (2-4 psi) above atmosphere to cool the rings rapidly.

7.12 Allow the heater-ring to cool sufficiently to permit handling (nominally 2 hours to reach 50°C). Then turn off the collector-plate heat exchangers, return the vacuum chamber to room pressure using the clean, dry nitrogen, and open the chamber.

7.13 Store nickel boats with specimens and respective collector-plates in desiccators (using silica gel desiccant) immediately. After specimens have cooled to approximately room temperature, but no longer than half-hour, remove and weigh each specimen within 2 minutes of its removal from the desiccators (M_f). Weigh the collectors (M_p'). Control collector-plates are used to detect cross-contamination or poor technique. The mass change of all the sample collector-plates must be corrected by subtracting or adding the average mass change of the three blanks before calculating the CVCM (M_b). A change in mass greater than ± 30 µg of any one of the blank collectors shall be cause for rejection of the whole run. The system must be thoroughly cleaned and out-baked prior to future use. All data acquired during runs when this occurs shall be discarded or retained with a note indicating the discrepancy.

7.14 Post-condition the foregoing samples at a relative humidity of 65% at 20°C for 24 hours to determine the RML. Re-weigh the post-conditioned specimens and boat (m_R).

8 Calculation of results

Three results are calculated:

(i) Total mass loss (TML).
(ii) Recovered mass loss (RML).
(iii) Collected volatile condensible material (CVCM).

The equations for these are:

(i) $$TML = \frac{(m_o - m_f) \times 100\%}{m_m}$$

(ii) $$RML = \frac{(m_o - m_R) \times 100\%}{M_m}$$

(iii) $$CVCM = \frac{(mp' - m_p \pm m_b) \times 100\%}{m_m}$$

where m_o = specimen + boat mass after pre-conditioning
m_f = mass of specimen + boat after test
m_m = mass of sample $(M_o - M_c)$
m_R = mass of specimen + boat after 24 hours post-conditioning
m_p = mass of collector before test
mp' = mass of collector after test
m_c = mass of sample boat
m_b = average mass change of three blank collectors.

9 Report

The report shall contain the following information:

(i) Trade name and number of the material, the manufacturer, the batch or lot number, or other such identification.

(ii) Summary of the preparation schedule (cure time and temperature, post-cure, cleaning procedures), date prepared.

(iii) Percentage of total mass loss, TML (the average value).

(iv) Percentage of recovered mass loss RML (the average value).

(v) Percentage of collected volatile condensable material, CVCM (the average value).

(vi) Infra-red spectrum or other analytical description of the condensed contamination when determined.

(vii) Remarks about any noticeable incident or deviation from standard conditions observed during the test.

10 Tolerances

The following tolerances shall apply:

All temperatures shall be $\pm 1^{\circ}C$ (unless otherwise stated).
Relative humidity ±5%.
Weighings ±1 µg.

11 General cleanliness and handling of samples

The weight changes being measured are extremely small, so it is imperative that samples are handled correctly to eliminate contamination and erroneous results.

General

(i) When handling parts of the apparatus within the vacuum chamber, clean nylon gloves must be worn.

(ii) Sample boats must only be handled with clean forceps or clean nylon gloves.

(iii) Collectors must only be handled using a suitable metallic handling device.

(iv) When preparing samples, clean nylon gloves must be worn.

Apparatus preparation

Sample boats

(i) Clean ultrasonically in a chloroform: acetone (50:50) mixture for 30 minutes.

(ii) Bake overnight in a vacuum oven at 165°C nominal and <50 mbar.

(iii) Store in a clean container, open to the atmosphere, at 65% relative humidity and 20°C.

Collectors

The procedure is similar to that described above, except that the collectors are stored for 24 hours in a desiccator after removal from the oven. When not in use the collectors shall be stored in a desiccator.

Apparatus

Both parts of the cooled ring should be swabbed with copious amounts of chloroform: acetone mixture, to remove condensed volatiles. Use a lint free cloth or white tissue.

The base plate around ports and ring supports should be swabbed to remove any dust.

Bake out of system

Prior to every run the system shall be baked out by heating the rings to a nominal 165° reducing the pressure to approximately 10^{-3} torr for a period of 16 hours.

Table 802.1

Test apparatus dimensions
(see Fig 802.1)

Letter	mm	Tolerance	Notes
A[A]	6.3	±0.1	diameter[B]
B[A]	11.1	±0.1	diameter[B]
C[A]	33.0	±0.1	diameter[B]
D[A,C]	13.45	±0.10	
E[A,C]	9.65	±0.10	
F[A,C]	0.65	±0.10	
G[C]	7.1	±0.3	
H[A]	0.75	±0.10	stock size
J[A]	12.7	±0.3	
K	9.6	±0.8	
M	16.0	±0.1	

For a system where compartments are arranged on a ring each individual chamber shall be at 50 ± 0.8mm centres and there shall be 24 compartments on each ring.

A Critical dimensions that must be maintained for test results to be comparable.

B Diameters must be concentric to ±0.1 mm for test results to be comparable.

C Dimensions include plating thickness. Satisfactory surfaces have been produced by making substrate surface finish 1.6 µm RMS highly polished, plated with electroless nickel, 0.0127 mm thick, and finished with electroplated chromium, 0.0051 mm thick.

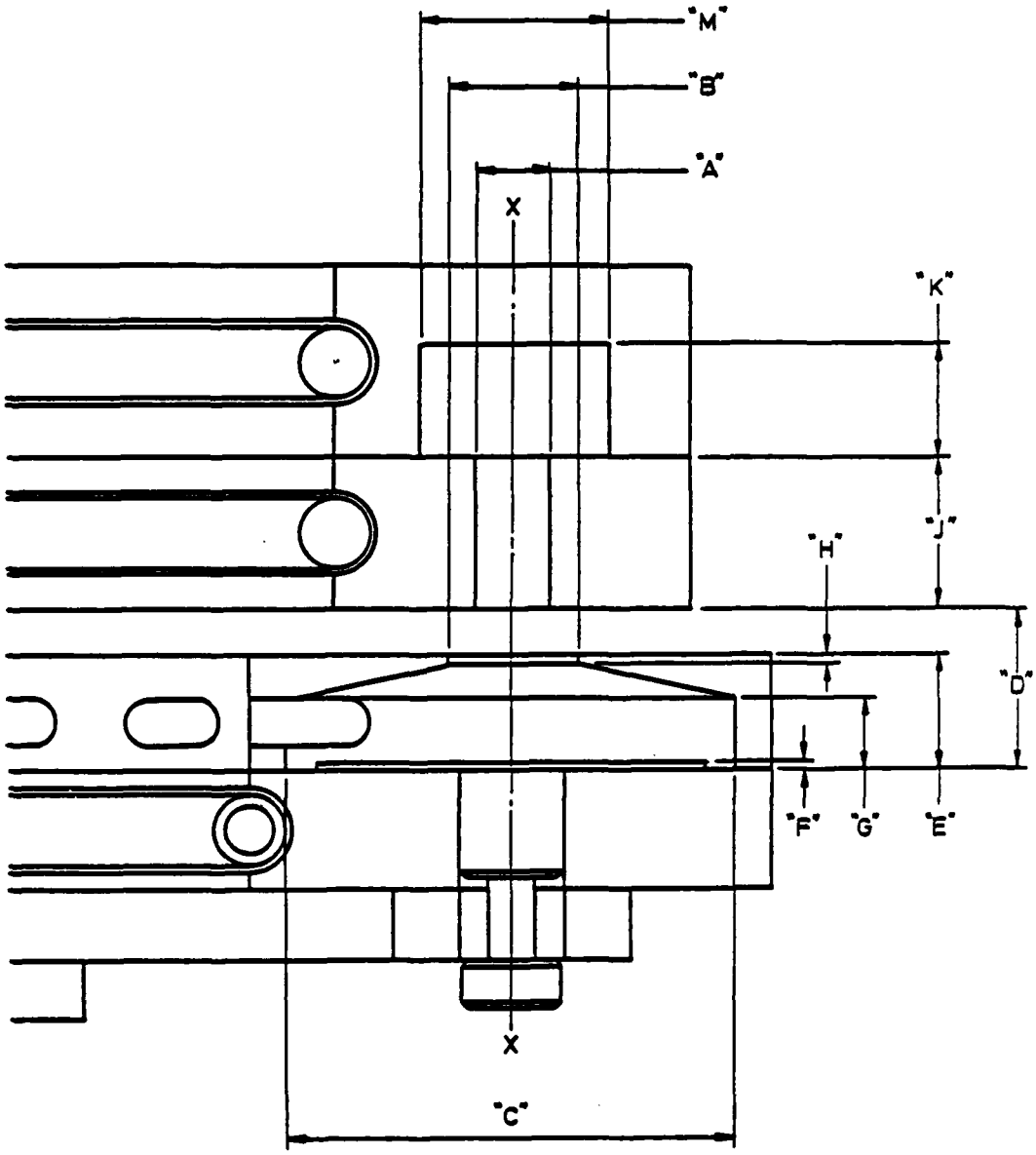

Fig 802.1 Apparatus
(for section through X-X see Fig 802.2)

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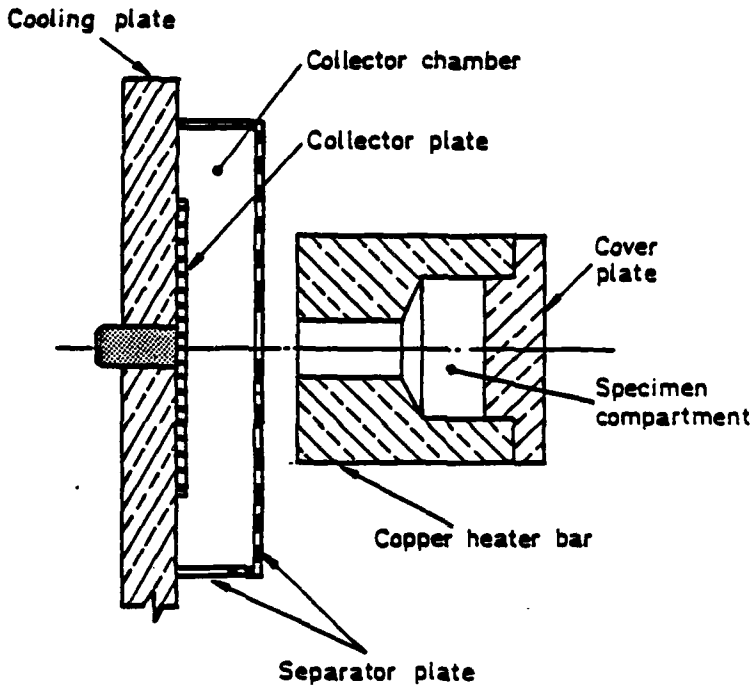

Fig 802.2 Schematic of critical portion of test apparatus (section X-X of Fig 802.1)

900 BACKGROUND INFORMATION ON ENVIRONMENTAL EFFECTS

1 Introduction

This sheet provides supplementary background information on environmental effects and is intended to be read in conjunction with the data sheets giving recommendations for the monitoring of moisture content[1] and for the conditioning of test specimens[2].

2 Effect of volume fraction

Except for the case of some polymer fibres, *eg* Aramid (see section 4) only the resin matrix absorbs moisture. Hence, M , expressed as a percentage of the composite weight, will decrease with increasing fibre volume fraction V_f .

Example 2.1

If at 60% V_f a composite contains 1.1% of water what is the expected water content at 70% V_f?

The following densities are assumed:

resin 1.3 gm/cc
fibre 1.8 gm/cc.

Assume a dry volume of 100 cc. With 60% V_f this gives 60 cc fibre and 40 cc resin. With the above densities this gives 108 gm, fibre and 52 gm resin, total 160 gm. For 1.5% moisture this gives 1.76 gm of absorbed moisture. Hence, as all moisture is held in the resin,

$$\text{Resin moisture content} = \frac{1.76}{52} \times 100 = 3.385\% .$$

With the same dry volume and material densities, but 70% V_f we have 126 gm fibre 39 gm resin and a total dry weight of 165 gm. Resin moisture content as a percentage will, however, be unchanged, hence

$$\text{Moisture content} = 39 \times 0.03385 = 1.32 \text{ gm}$$

and

$$\text{Composite moisture content} = \frac{1.32}{165} = 0.8\% .$$

3 Effect of voids

Generally, the material will be rejected if the void content V_v is significant and the value of 2% used in this example must be considered as high. A distinction is made between 'external' voids where moisture can enter without

passing through any part of the resin matrix and 'internal' voids where at least some diffusion through the resin must take place.

In the case of internal voids the effect of the moisture within the void on the measurement of moisture uptake would appear to be small. Following Ref 3, we can expect for initially dry material that the relative humidity within the void will rise to eventually reach the conditioning humidity. Even at 100% RH figures given in Ref 4 show that, at 60°C, the mass of the moisture in the voids for 2% V_v is only about 2×10^{-7} of the specimen mass. The problem here, which is considered in Ref 3, is the effect of a rise in temperature.

For external voids the extreme case to be considered is when these voids are filled with water. Clearly, it is not possible to remove this prior to weighing. The following example considers such a case, with V_v = 2%, and all voids of the external type filled with water.

Example 3.1

If a 60% V_f composite with a void content V_v of 2%, all of the external type and full of water, contains 3% of moisture, at saturation, what is the expected saturated moisture content of a void free laminate of the same V_f ?

Assuming a dry volume of 100 cc, this gives 58.8 cc fibre, 39.2 cc resin and 2 cc void. With the same densities as in the example of section 2.8, this gives a total weight of 156.8 gm, with 50.96 gm of resin. The total moisture content is therefore 4.704 gm, 2 gm in the voids and 2.704 gm in the resin, hence

$$\text{Resin moisture content} = \frac{2.704}{50.96} \times 100 = 5.306\% \ .$$

For a dry void free volume of 100 cc with 60% V_f, we have 60 cc fibre and 40 cc resin, giving 108 gm fibre and 52 gm resin, a total dry weight of 160 gm. Resin moisture content as a percentage will however be unchanged, hence,

$$\text{Moisture content} = 52 \times 0.05306 = 2.759 \text{ gm}$$

and

$$\text{Composite moisture content} = \frac{2.759}{160} = 1.724\% \ .$$

4 Moisture uptake of glass fibre and polymer fibre composites (*eg* aramid)

Although not quite as important with the new conditioning methods, it is nevertheless useful to know how to estimate the expected moisture uptake of one type of composite from data on another type. In this section this is done for glass fibre and aramid composites using data obtained from CFC.

Example 4.1

Using the following data, what moisture level is expected in a glass fibre composite at 70% RH?

Density of resin = 1.3 gm/cc
Density of glass fibre = 2.55 gm/cc
Moisture level in resin at 70% RH = 3.434%
Volume fraction = 60%.

Assume a dry volume of 100 cc. With 60% V_f this gives 60 cc fibre and 40 cc resin. With the above densities this gives 153 gm fibre and 52 gm resin, total 205 gm. Hence, as glass fibre does not absorb moisture,

Mass of absorbed moisture = $52 \times 0.03434 = 1.786$ gm

Hence moisture update of glass fibre composite = $\frac{1.786}{205} = 0.87\%$.

Example 4.2

Using the following data, what moisture level is expected in an aramid fibre composite at 70% RH?

Density of resin = 1.3 gm/cc
Density of aramid fibre = 1.45 gm/cc
Moisture level in resin at 70% RH = 3.409%
Moisture level in fibre at 70% RH = 3.0%
Volume fraction = 60%.

Again, assume a dry volume of 100 cc. With 60% V_f and the above densities this gives 87 gm fibre and 52 gm resin, total 139 gm resin. Aramid fibres are moisture absorbent, hence

Mass of moisture absorbed into fibres = $87 \times 0.03 = 2.61$ gm

Mass of moisture absorbed into resin = $52 \times 0.03409 = 1.77$ gm

Total absorbed moisture = 4.38 gm

Hence moisture uptake of aramid composite = $\frac{4.38}{139}$ = 3.15% .

Comparative figures

Table 900.1 gives a comparison of expected moisture levels in carbon fibre, glass fibre and aramid composites at three levels of humidity, 70%, 85% and 95%. Note that for the glass fibre composite the resin properties assumed are taken from data for a 125° cure system. For the carbon and aramid composites the properties are taken from data for a 180°C cure system. Please note also the remarks of section 6.

5 Loss of moisture during elevated temperature testing

As pointed out in Ref 2, testing at elevated temperature can involve significant moisture loss. This applies particularly to fatigue testing where extended elapsed times are generally the rule and to full scale structural or large structural element tests where only fairly slow heat-up rates can be achieved.

For coupon testing it has been common practice to allow a 10 minute 'heat soak' to take place at the test temperature prior to the commencement of loading. The purpose of this soak is to eliminate distortion due to non-uniform temperature distributions.

It is clearly undesirable that this drying should take place as at least partial recovery of property can be expected in the affected outer plies. For large scale tests, including fatigue tests, it is strongly advised that moisture levels be maintained during the loading sequence. This should be done by maintaining the appropriate humidity level throughout and may require the construction of an *ad hoc* climate chamber round the test piece. If the test temperature is in excess of 100°C high humidity levels can be maintained only by raising the boiling point by pressurisation ('super heated' steam[5])

Testing performed at BAe[6] has shown that for 3mm thick FADD specimens the heat soak time can be reduced to 2-3 minutes. This could be employed for most coupon tests although for thicker specimens and possibly for different test rigs somewhat longer soaks may be necessary. Subject to this proviso, it is recommended that heat soak times be reduced to this lower level.

In order to illustrate the effect of drying during elevated temperature some simple calculations have been performed. Taking 2mm thick XAS/914C material as an example, it has been assumed that conditioning was to moisture level of

1.4% (95% of saturation) at a cabinet setting of 85% RH. It was then assumed that the material was dried at 120°C, with zero surface humidity. The moisture gradients calculated after drying periods of 3, 10, 60 and 120 minutes respectively, are illustrated in Fig 900.1. Even with 120 minutes heat soak only the outermost 3 plies are significantly affected, but in these plies the drying is considerable and appreciable recovery of material property is to be expected. Against this there would be some risk of damage to the material at this temperature but the recovery effect is expected to predominate. Normal Fickian theory[7] has been used in these calculations.

Table 900.1

RH	Expected moisture level for fibre type - %		
	Carbon	Glass	Aramid
70%	1.11	0.87	2.15
85%	1.47	1.27	4.32
95%	1.78	1.58	5.38

The effect on failure load of the near surface moisture gradients shown in Fig 900.1, clearly depends on the nature of the loading. Fibre dominated properties will be little affected. The same may well be the case for matrix dominated properties such as ud compression. Under uniform moisture gradient each ply takes an equal share of the load and under ideal conditions failure would be virtually simultaneous in each ply. Because the compression modulus is fibre dominated this equality of loading in each ply will remain despite the recovery of strength in the outer plies. Failure is therefore expected to occur in the interior plies unaffected by the drying at more or less the same level as for undried material.

However, in the case of ud material under flexure, failure is expected in compression in the outermost plies where the stress levels are highest. As these outer plies dry out and recover strength the point of failure can be expected to move inwards and the failure load will rise as a result as the critical point moves closer to the neutral axis.

Table 900.2 illustrates this recovery of property through drying of the outer layers. Based on the simplistic use of a laminate strength prediction

method[8], it deals with pure M_x bending loading. Failure levels of M_x are quoted for drying periods of 0, 3, 10, 60 and 120 minutes.

Table 900.2

Effect of drying prior to and/or during testing on XAS/914C failure level at 120°C under M_x bending load

Predicted failure level of M_x (Nmm per mm width) after drying at 120°C for				
0 min	3 min	10 min	60 min	120 min
505	540	577	666	716

6 Notes on examples

All examples and figures given in sections 2 to 5 are for guidance only. Users needing to make similar calculations should make sure they have the correct data appropriate to the particular fibre and resin systems with which they are concerned.

7 Physical ageing

Physical ageing of composites is discussed in Ref 9. This phenomenon generally leads to the moisture content at saturation at a given humidity level reducing somewhat as elapsed time since material manufacture increases. It is one of the reasons why it has been considered desirable to condition to saturation at an appropriate humidity level rather than to a target value of moisture uptake.

It is, however, not yet established that the ageing rates of resin materials are sensibly constant even within a particular family. More precise information is required before guidelines can be given on the reduction of the saturation moisture content with age.

8 Langmuir effects

The so-called Langmuir effect only becomes significant at large exposure times, as a slow upwards creep of the moisture content at times greater than those at which the Fickian theory predicts saturation. A theoretical model for this phenomenon has been put forward[10]. Using parameters used in Ref 10 for neat 5208 resin at room temperature and 75% RH Fig 900.2 has been derived comparing Langmuir and Fickian predictions. The specimen size was 25.4 × 12.7 × 0.86 mm.

From Fig 900.1 it will be observed that:

(a) The Langmuir and Fickian models differ significantly only at large exposure times. (Note that 100 days exposure for 0.86 mm thickness gives roughly the same moisture uptake as 546 days for 2 mm thickness).

(b) The Langmuir and physical ageing effects appear to be in opposition. However, the Langmuir effect manifests itself during a continuing absorption process. Physical ageing appears to occur mainly during storage dwells.

(c) The Langmuir effect appears to make more difficult the determination of the equilibrium concentration.

9 Note on quick methods

The recommended conditioning method[1,2] involves longer conditioning times than previous techniques where material was soaked to a target value of moisture content using a high humidity. Consequently, and in particular where thicker material is involved the pressure to resort to 'quick' methods is difficult to resist.

The design of such quick methods, if they are to give any reliable information at all, requires some rough knowledge of the material diffusion parameters. Even where adequate information is available the conditioning can only be shortened by reverting to earlier unsatisfactory methods. Either an unacceptably high temperature must be used or an unrepresentative moisture gradient produced.

It is in fact possible to reduce the conditioning times under the recommended method by using a multi-stage conditioning technique such as those described in Ref 12. In such cases the humidity setting of the final stage has to be the recommended value (*eg* 85% for the world-wide case), but higher humidities can be used in the earlier stages. The optimum multi-stage regime will depend on the particular application, especially if the test piece has more than one critical thickness. These reductions in timescales, though worthwhile, fall far short of bringing the new regimes into the old order of project timescales, achieved however by means now considered to be unacceptable.

While recognising that project timescales may force compromises in certain cases, it is not possible to make recommendations on quick methods in view of the known disadvantages[9,11].

10 References

1 CRAG W.G. on Test Methods. Method 901. Recommendations for the determination of diffusivity properties.

2 CRAG W.G. on Test Methods. Method 902. Recommendations for the conditioning of test specimens.

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9 Environmental effects on the testing of composite structures in aircraft, Report to CRAG. Ref. BAe-MSM-R-GEN-0603, June 1985.

10 H.G. Carter and H.G. Kibler. Langmuir-type model for anomalous moisture diffusion in composite resins. J. Comp. Mat (12), April 1978.

11 T.A. Collings. RAE Technical Memorandum Mat/Str 1034, February 1984.

12 S.M. Copley and T.A. Collings. The determination of through-thickness moisture distribution and diffusion coefficients in fibre reinforced plastic laminates. RAE Technical Report 81105, August 1981.

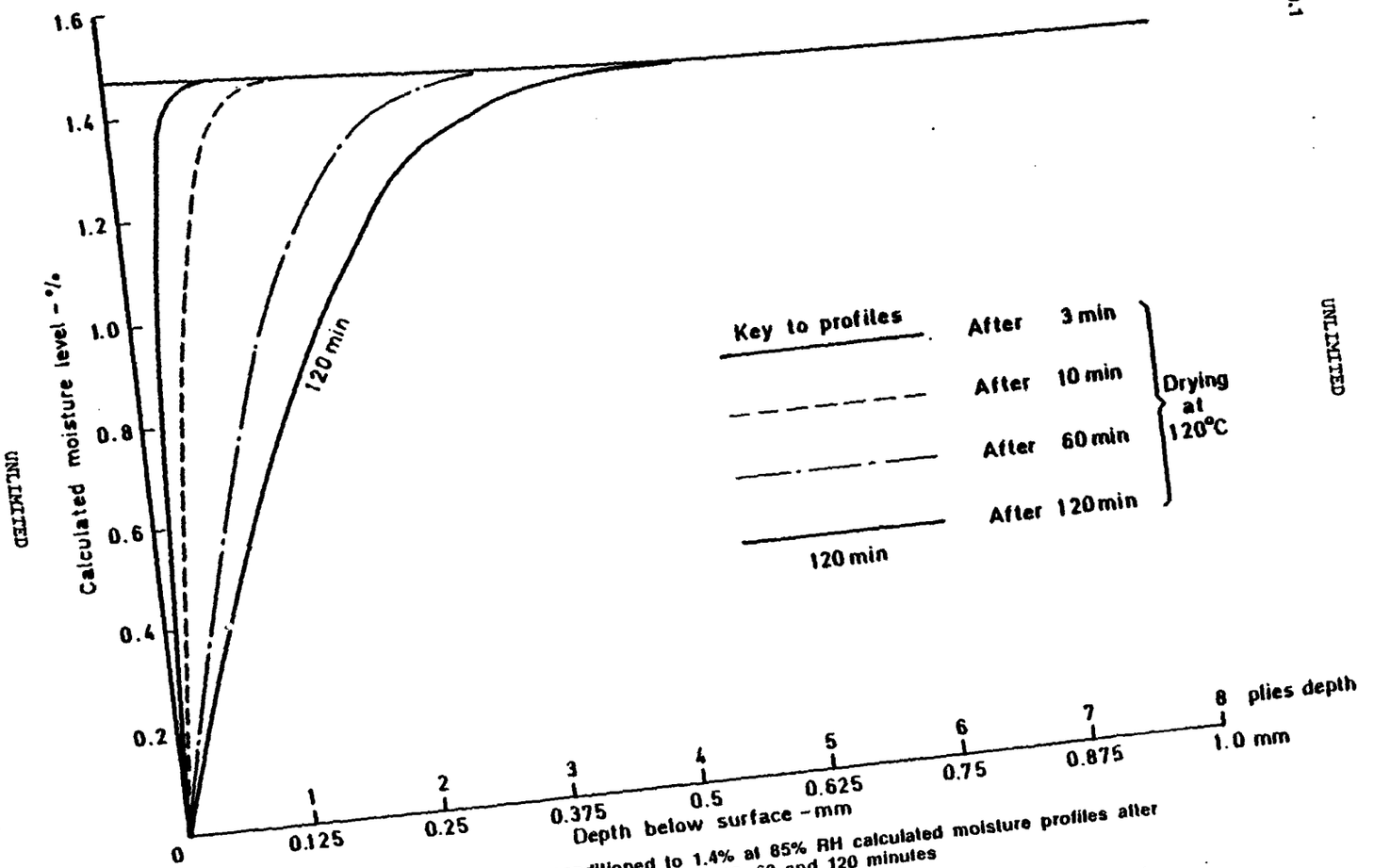

Fig 900.1 2 mm thickness conditioned to 1.4% at 85% RH calculated moisture profiles after drying at 0% RH and 120°C for 3, 10, 60 and 120 minutes

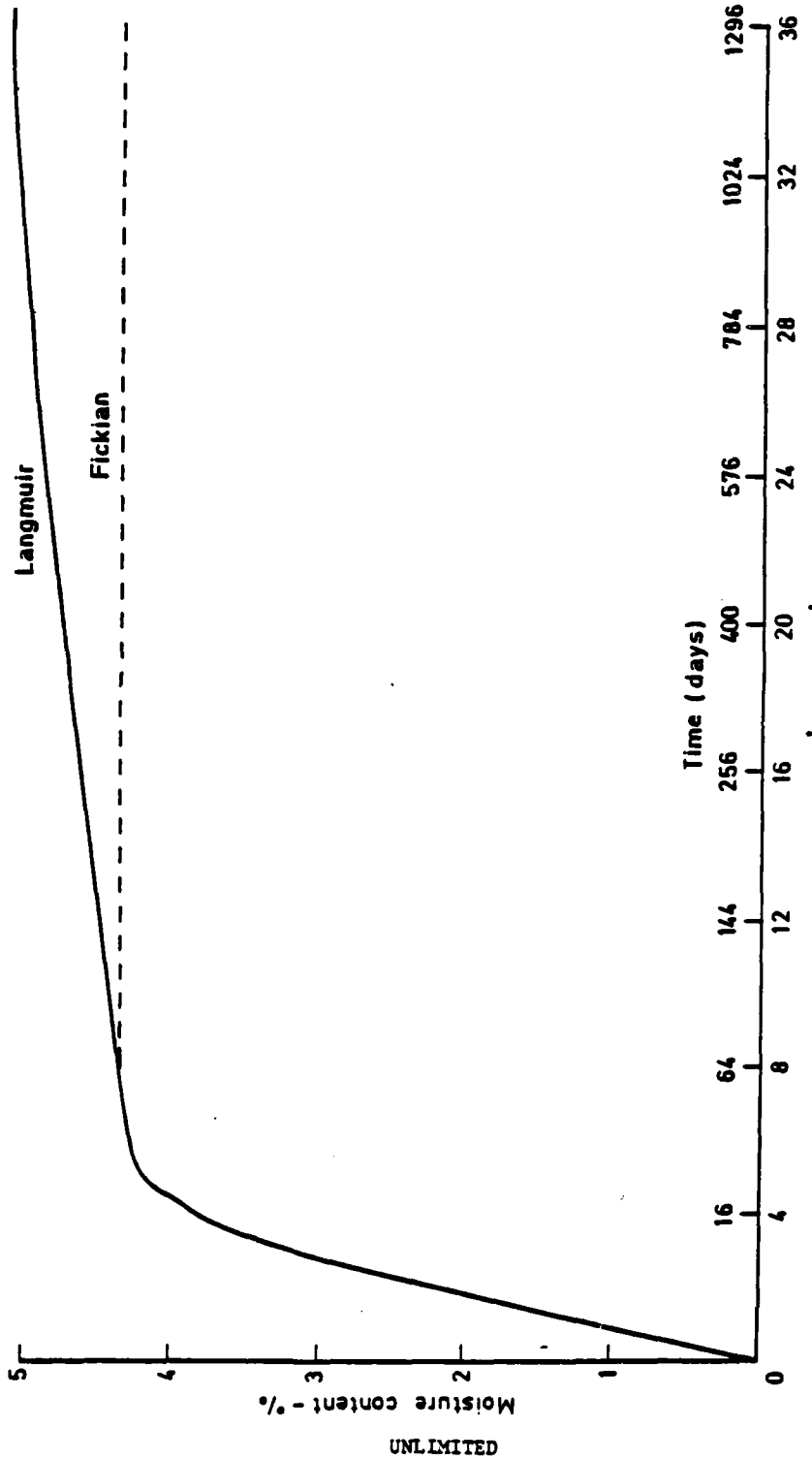

Fig 900.2 Comparison of Fickian and Langmuir models

901 METHOD OF ASSESSMENT OF DIFFUSIVITY PROPERTIES OF FIBRE REINFORCED PLASTICS

1 Description

This data sheet contains recommendations for the determination of the moisture diffusivity characteristics of a given material.

These recommendations are not restricted to any particular material or generation of materials, except in as much as the limits quoted on the conditioning and drying temperatures may have to be altered for the new developing resin systems. Unless convincing evidence to the contrary is produced hence or otherwise it is suggested that the guidelines evolved for the present generation of resins be retained until the necessary data is generated.

2 Method

2.1 Specimen size and sample size

Because the actual moisture content is never more than a few percent of the total weight, it is clearly undesirable from an accuracy viewpoint to monitor it using small coupons such as the ILSS specimen. The recommended number of plies is that which gives a nominal thickness of 2 mm (this would be 16 for a ply thickness of 0.125 mm). 75 mm is recommended for both length and width and the specimen is illustrated in Fig 901.1. The large length/width to thickness ratio is also valuable in reducing the effect of diffusion in directions other than through the thickness (edge effects). A tolerance of ±1 mm on length/width and ±0.2 mm on thickness is allowable.

Applications where the diffusion coefficients in other directions are required are regarded as special cases and outside the scope of this data sheet.

A sample size of 3 is recommended.

2.2 Pre-experimental exposure

Moisture is readily taken up by dry laminates when they are exposed to the laboratory atmosphere. Diffusion parameters deduced from tests where the pre-experimental exposure is significant may be appreciably in error[1] and every effort should be made to minimise this parameter. Vacuum storage or sealing in metalised polythene or aluminium bags is recommended.

It is mandatory to determine the zero weight datum. In order to determine this the specimens should be dried to constant weight, immediately prior to the commencement of exposure, irrespective of the method of storage employed.

2.3 Weighing procedure and moisture uptake determination

Weighings should always be made as soon as possible following withdrawal from the conditioning chamber/oven. However, it is essential to remove any surface moisture using a dry cloth or tissue. The balance doors should be closed while weighing and long tweezers used to handle the specimens. Draughts and body heat effects can induce appreciable distortion into the readings. The balance should be capable of measuring to ±0001 gm. It is important that specimens and travellers spend the minimum time outside the conditioning environment and that the conditioning chamber is managed so that it is opened for the minimum time possible on the minimum number of occasions.

Knowing both the wet and dry weights w_w and w_d respectively, the percentage uptake of water by weight, M , can be found from

$$M = \frac{(w_w - w_d)}{w_d} \times 100\% \quad . \tag{1}$$

2.4 Conditioning and drying temperatures

These recommendations stem from Ref 2 and are based on the principle that a transition temperature appears to exist for all resins at higher values than which non-Fickian absorption/desorption behaviour is observed. It is not therefore recommended that these temperatures be exceeded unless convincing evidence has been obtained in justification.

The recommended maximum conditioning temperatures are 70°C for 180°C cure systems and 45°C for 120°C cure systems. For drying it is recommended that a vacuum oven be used set no higher than 70°C.

For some new materials the temperatures recommended may be too high, while for others designed for improved elevated temperature properties higher conditioning temperatures may be acceptable. In order to investigate these possibilities it is suggested that a pilot experiment be carried out using a single specimen of dimensions 50 × 50 × t mm where t represents the thickness (0.5 mm nom) of a 4-ply 0/90 symmetric lay-up.

Starting at 70°C, the pilot experiment should be repeated at temperatures lower by 5°C (*ie* 70°C, 65°C, 60°, etc) until Fickian behaviour is observed. Fig 2 illustrates a typical Fickian plot of moisture against the square root of time. If Fickian behaviour is observed at 70°C the same procedure can be followed at successively higher temperatures (*ie* 70°C, 75°C, etc) until non-Fickian behaviour is produced. For 120°C cure materials the starting temperature should be 45°C. However, some apparent deviations from Fickian behaviour may arise merely from the small net weight and rapid approach to saturation of the pilot specimen.

2.5 Test matrix (including lay-ups)

It is recommended that the same tests are carried out using u/d and 0°/90° symmetric lay-ups.

It is recommended that the absorptivity be determined at a minimum of three different relative humidities, *eg* 70%, 85% and 95%. The acceptable tolerance on humidity is +5%. The tolerance on temperature is ±2°C. In order to explore the relationship between absorptivity and relative humidity, it may be required to perform experiments at a number of other humidity levels. The use of either conditioning cabinets or salt solutions is acceptable provided the tolerances on humidity and temperature are maintained throughout. Similarly in order to explore the relationship between temperature and diffusivity it may be required to perform experiments at a number of temperatures below the recommended maximum.

2.6 Frequency of weighings

Fig 901.2 shows a typical Fickian plot of moisture content against the square root of time. It will be seen that the rate of moisture uptake decreases fairly rapidly with time, so that a higher frequency of weighings is required in the early stages.

It is recommended that if possible the experiment commences on a Monday morning. At least three weighings should be made on the first day and at least two during the remainder of the first week. At least one weighing should be made each day during the second week, followed by a gradual decrease in frequency as the rate of weight increase diminishes.

2.7 Determination of 'levelling-off' and of diffusion parameters

The determination of the equilibrium concentration, represented by the height of the flat portion of Fig 901.2 at large values of time,

requires a definition of 'levelling-off' in terms of permissible difference between maximum and minimum moisture content measurements over a given period of time. A similarly shaped curve showing weight loss against the square root of time results from drying out.

It is recommended that levelling-off be defined as no greater variation than ±0.5% in moisture content (*ie* ±0.005% of composite weight for a saturation level of 1.0% moisture) over a period of time which is a function of conditioning temperature and material thickness and during which at least three weighings should be made.

For 180°C cure systems it is recommended that at 70°C and for 2 mm thickness this period should be three weeks. For other thicknesses and conditioning temperatures the recommended period is given by:-

$$W = \frac{t^2}{154210 \exp\left(\frac{-4000}{T}\right)} \qquad (2)$$

where W = dwell time in weeks
t = specimen thickness (mm)
T = absolute conditioning temperature (°K).

For 120°C cure systems it is recommended that at 45°C and for 2 mm thickness the period should be 3 weeks. The corresponding formula for other thicknesses and conditioning temperatures is:-

$$W = \frac{t^2}{204300 \exp\left(\frac{-3798}{T}\right)} \; . \qquad (3)$$

The diffusion coefficient is determined from the straight line portion of Fig 901.2 at the lower values of time. Following Ref 3 the diffusion coefficient, D, is given by:-

$$D = \frac{\pi}{16}\left(\frac{t(M_2 - M_1)}{M_\infty(\sqrt{T_2} - \sqrt{T_1})}\right)^2 \; , \qquad (4)$$

where M_∞ = equilibrium concentration
M_1 = moisture uptake after time T_1
M_2 = moisture uptake after time T_2
t = thickness.

To give a better estimate of the one-dimensional diffusion coefficient D_∞, a correction factor given by Shen and Springer[4] can be used giving:-

$$D_\infty = \frac{D}{\left(1 + \frac{t}{w} + \frac{t}{l}\right)^2} \quad . \tag{5}$$

2.8 Measurement of moisture gradients

The moisture gradient through-the-thickness can be measured by using the slicing technique described in Ref 3. By this means the time required to determine M can be shortened as the outer plies will always saturate first.

2.9 Volume fraction

It is recommended that the fibre volume fraction be as near to 60% as possible. Results obtained using material of different volume fractions can, however, be normalised to a 60% V_f value using the method given in Ref 5.

3 Test report

The test report should include the following particulars:-

(a) Complete identification of the material tested, including fibre and resin type, date and method of manufacture, lay-up, stacking sequence, batch number, void content, volume fraction and any previous history.

(b) Sample dimensions.

(c) A complete tabular and graphical representation of all the experimental sampling, including any pilot experiments.

(d) The diffusion parameters D and M_∞ deduced from the test results.

(e) Date of commencement and conclusion of test.

4 References

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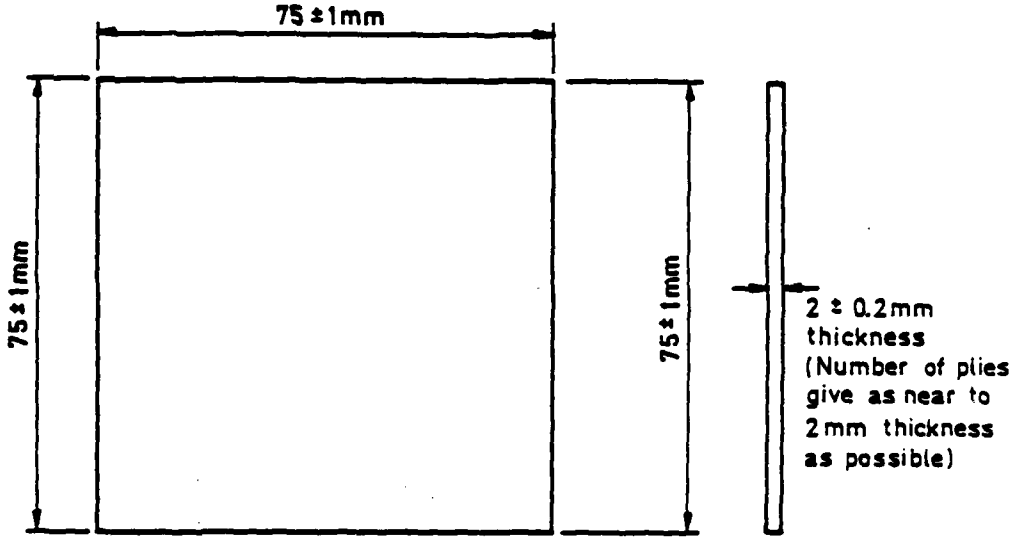

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Fig 901.1 Recommended test specimen for the determination of diffusion parameters

Fig 901.2

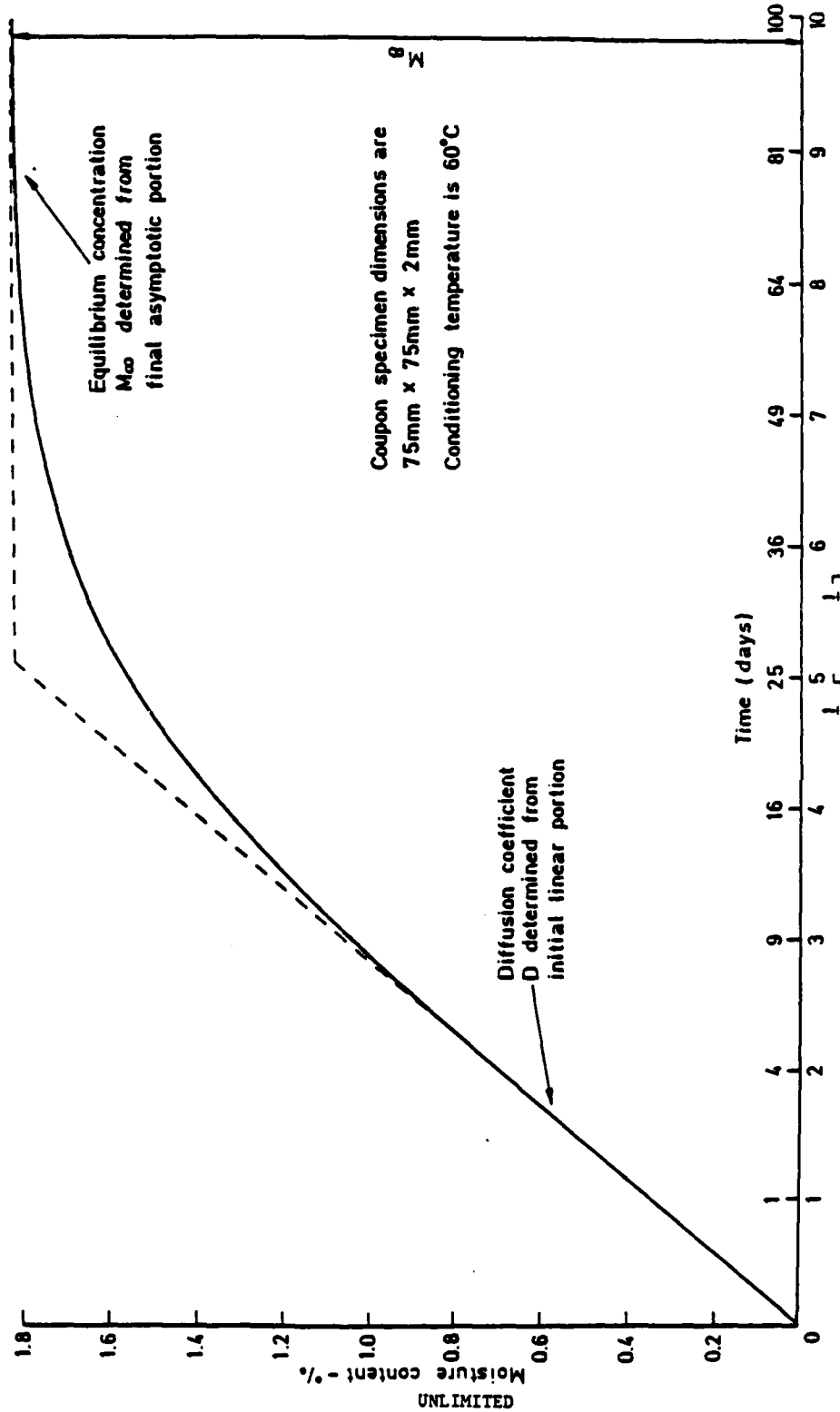


Fig 901.2 Fickian diffusion curve

902 METHOD OF CONDITIONING OF FIBRE REINFORCED PLASTICS UNDER HOT/WET ENVIRONMENTS

1 General

For the purpose of this method it is assumed that test specimens will be coupons or simple structural elements of uniform thickness (1-4 mm) which will be used to establish mechanical behaviour over a range of uniform moisture contents and temperature levels. Situations involving changes in thickness, built up structure and testing involving moisture gradients will need more complex strategies and the reader is referred to Ref 1, where moisture absorption is discussed in more detail. It is assumed that moisture will be gained or lost through all surfaces of the test section and that moisture content will be determined by weight changes.

Specimens may be fitted with end tabs involving the use of other materials and adhesives. Some testing will be long term and may involve elevated temperature levels. Temperature differences may exist between the test section and its ends gripped in testing machines. Damage may accumulate during long term test sequences. These factors make the monitoring of moisture content by the weighing of specimens inaccurate and inconvenient and the use of travellers for this purpose is now widely accepted.

2 Traveller requirements

Travellers are required to monitor specimen moisture content by weighing. They should be cut from the same board adjacent to the specimen involved and will therefore be of the same lay-up, thickness, batch and have experienced the same processing sequence. Traveller width and length should be comparable with the specimen test section involved, provided that an accuracy in moisture content determination of ±1% can be obtained. Where this is not the case (in small or thin specimens) the traveller width and length should be increased. For specimen thicknesses of 1 and 2 mm, travellers of 45 × 45 mm and 30 × 30 mm respectively are recommended. Each specimen should be provided with its own traveller which should accompany it at all times throughout its environmental history of manufacture, storage, pre-conditioning and testing so that weight changes may be monitored.

Composite material will absorb moisture from the environment as soon as it is exposed after processing. To limit this, storage in vacuum, at room temperature, of material, specimens and travellers is recommended. Alternatively bagging in sealed impervious bags containing desiccant may be adopted. Even so

some moisture absorption can be anticipated and a moisture content datum must be established. It is therefore recommended that additional secondary travellers be provided to establish the moisture content at the start of the pre-conditioning phase by drying them in a vacuum oven at that time to constant weight at an approved temperature level (see section 4).

It should be noted that where specimens are required for testing in the 'as received' or the dry condition secondary travellers will not be required.

3 Pre-conditioning

Current policy is to pre-condition to equilibrium at a constant relative humidity (RH) equivalent to overall service usage in the environment of interest. Comparisons between material systems with differing moisture absorption characteristics are then possible since each would arrive at its own moisture content appropriate to a common sequence of usage. A RH of 85% is recommended to represent the worst world-wide average usage while the dry condition, although not representative of service conditions forms a convenient datum level which is widely used. Intermediate levels may be required for further material characterisation and it is recommended that these should be representative of actual usage. 70% RH has been used for temperate environments while desert conditions at low RH levels may also be of interest.

4 Temperature levels for drying and pre-conditioning

Drying and pre-conditioning may be accelerated by raising the temperature level but care is necessary since damage may be produced if temperature levels are too high (see method 901). Ref 1 recommends maximum temperature levels of 60°C and 45°C for current thermosetting materials processed at 180°C and 120°C respectively. Other material systems may have different limits but the temperature levels quoted above should not be exceeded unless sufficient evidence has been obtained to justify it.

5 Monitoring of drying and pre-conditioning

It is recommended that both specimens and travellers be removed from the conditioning environment when travellers are weighed to determine moisture content. Surfaces should be wiped using a dry cloth or tissue and travellers should be weighed, after cooling to room temperature, using an analytical balance capable of measuring to 0.0001 gm. Balance doors should be closed during weighing and travellers should be handled using long tweezers. Weighing should be carried out at approximately equal intervals of $\sqrt{\text{time}}$, (see method 901). Conditioning

to the 100% equilibrium moisture content and its confirmation is costly in terms of time and it is therefore recommended that the 95% level should be adopted based on three successive measurements exceeding this value. It should be noted that this requirement implies knowledge of the 100% level either from initial experimental work on identical travellers or from a prediction, based on a diffusion coefficient derived from initial moisture uptake and subsequently confirmed experimentally. It is important that specimens and travellers spend the minimum time outside the conditioning environment and that the conditioning chamber is managed so that it is opened for the minimum time possible on the minimum number of occasions.

Where both primary and secondary travellers are involved, simultaneous moisture conditoning and drying will be necessary. During these procedures the percentage moisture (M), loss or gain, can be monitored relative to the common starting condition as follows:-

$$M = \frac{(W_2 - W_1)}{W_1} \times 100\%$$

where W_1 is the weight at the start and W_2 is the current weight of the traveller (primary or secondary) being recorded. When the secondary traveller has been completely dried the initial percentage moisture content will be known and may then be used to calculate the dry weight of the prime traveller. The total percentage moisture content (M_T), relative to the dry condition, can subsequently be found from:

$$M_T = \frac{(W_p - W_D)}{W_D} \times 100\%$$

where W_p is the current weight of the primary traveller and W_D is its calculated weight in the fully dried condition.

Where specimens are not immediately required for testing they may be stored in the pre-conditioning environment with routine weight monitoring until required. On removal for testing the traveller should accompany its specimen so that monitoring can continue throughout life.

When testing at elevated temperatures significant moisture loss can occur especially if an extended time period is involved and special measures may be required to maintain moisture content[1].

6 Pre-conditioning and test records

Recording of environmental history should include the following particulars.

(a) Material identification comprising fibre and resin type, lay-up, stacking sequence, batch number and previous history.

(b) Traveller and specimen identification, dimensions and fibre volume fraction.

(c) Pre-conditioning sequence including relative humidity, temperature and times.

(d) Tabulated, measured weights and derived moisture contents, dates, pre- and post-conditioning storage conditions.

(e) Mechanical test details including date, environmental sequence with times and pre- and post-test traveller weights and moisture contents.

(f) Traveller dry out details.

7 References

1 Environmental effects in the testing of composite structures in aircraft. Report to CRAG, Ref BAe-MSM-R-GEN-0603, June 1985.

1000 METHODS OF ASSESSMENT OF FIBRE VOLUME FRACTION OF FIBRE REINFORCED PLASTICS

Introduction

These methods describe the procedures to be used to determine the fibre volume fraction (Vf) and the resin volume fraction (Vr) of cured fibre reinforced plastic laminates.

It is not recommended that determination of the void content of laminates be undertaken using any of the methods detailed below (see, however, section 1001).

No one method is recommended in preference to any other. However, when quoting values of Vf and Vr the method of measurement should also be quoted.

The inaccuracies associated with each measurement are such that values of Vf and Vr should be quoted to the nearest 1% only.

1 'Density measurement' method

This method assumes negligible voidage in the laminate and hence should not be used when the level of voids is known to exceed 1%.

Determine the density of the composite (ρc) as described under section 800.

Prepare a linear graph of density versus per cent fibre content (see Fig 1000.1). Plot the resin density value at the per cent fibre = 0 value and plot the fibre density at the per cent fibre = 100% value. Join these two points with a straight line.

Draw a parallel to the per cent fibre axis (X-axis) from a point on the density axis (y-axis) to intersect the previously drawn line. From this point of intersection drop a perpendicular to intersect the per cent fibre axis thus giving an estimate of the volume fraction of the composite.

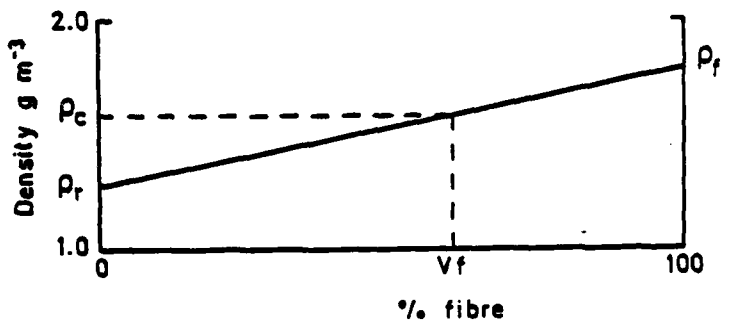


Fig 1000.1

012

where ρc = composite density g/m
ρf = fibre density g/m
ρr = resin density g/m.

NOTE: fibre and resin density should be obtained from the suppliers release documentation.

The resin volume fraction may be obtained from the following:-

$$Vr(\%) = 100 - Vf \ .$$

2 'Thickness measurement' method

Obtain the average thickness of the sample. At least five measurements should be made along the length of the sample. Correction may need to be made for excess surface resin, such as that introduced by a peel ply during manufacture.

The fibre volume fraction of the sample may be obtained from the following:-

$$Vf(\%) = \frac{N \times Mp}{t \times \rho f} 10^{-1}$$

where N = number of plies in the laminate
Mp = pre-impregnate fibre weight per unit area (g/m) (1)
t = laminate thickness (mm)
ρf = fibre density (g/m) (2)

NOTES: (1) The pre-impregnate fibre weight may be obtained from either the suppliers release documentation or by direct measurement from a sample taken from an area adjacent to that area of pre-impregnate used in the manufacture of the laminate.

(2) Fibre density should be obtained from the suppliers release documentation.

The resin volume fraction may be obtained from the following:-

$$Vr(\%) = 100 - Vf \ .$$

3 'Resin removal' method

Determine the density of the composite sample (ρc) as described under section 800.

Determine the mass of the composite sample (Mc).

Determine the mass of the fibre in the composite sample (Mf). This may be done in one of three ways depending upon the system being investigated.

(a) Sulphuric acid/hydrogen peroxide digestion

Place the specimen in a 500 ml conical beaker and add 20 ml of concentrated sulphuric acid. Gently heat the beaker until the acid begins to fume, then reduce heat and leave to digest for a few minutes (do not allow volume to become too small as bumping may occur). Carefully add 30 ml of 100 volume hydrogen peroxide, drop wise at first, faster when reaction is nearing completion. If solution is still brown, further 5 ml portions shall be added at one drop per second until the solution becomes clear.

Boil the resulting mixture for 10 minutes. Allow the solution to cool.

Weigh a clean, dry, scintered glass crucible (porosity 3) W1. Filter digestion mixture through the scintered glass crucible using a Buchner filtration flask and pump (take care not to loose any fibre), washing the beaker thoroughly with distilled water.

Wash fibre in scintered crucible until filtrate is neutral to universal indicator paper.

Use pump to remove as much liquid as possible, before drying the crucible plus fibre at 125-150°C for approximately 45 minutes. Care must be taken to avoid any loss of fibre.

Allow to cool to room temperature in desiccator then weigh the crucible plus contents W2.

(b) Nitric acid digestion

Place the specimen in a beaker containing at least 50ml of concentrated nitric acid.

Heat the acid to boiling point and hold for 60-90 minutes. Allow the solution to cool.

Weigh a clean, dry, scintered glass crucible (porosity 3) W1. Filter digestion mixture through the scintered glass crucible using a Buchner filtration flask and pump (take care not to loose any fibre), washing the beaker thoroughly with distilled water. Repeat this process this time washing the beaker thoroughly with acetone.

Proceed as in (a) above to obtain the weight of the crucible plus contents (W2).

To allow for any loss of fibre due to oxidation by the acid, a BLANK, comprising a known weight of fibre should follow an identical 'digestion' path to that defined in either (a) or (b) above. The percentage loss of weight may be defined as:

$$Mb = 100(W3 - W4)/W3$$

where W3 = original fibre weight (g)
W4 = final fibre weight after 'digestion' (g).

The mass of the fibre (corrected for any weight loss due to oxidation by the acid) is defined as:

$$Mf = (W2 - W1)/(100 - Mb) \ (g) \ .$$

(c) Resin burn-off

This method should not be used for CFRP.

Place the sample in an oven pre-heated to 580-600°C. Allow resin to burn-off for at least 1 hour or until all traces of blackness on the sample have disappeared. Remove the fibres from the oven and allow to cool to room temperature in a desiccator. Obtain the mass of the fibres (Mf).

Calculations

The fibre volume fraction can be calculated from the following:-

$$Vf(\%) = \frac{100Mf\rho c}{Mc \times \rho f} \ .$$

The resin volume fraction can be calculated from the following:-

$$Vr(\%) = \frac{100(1 - Mf)\rho c}{Mc \times \rho r} \ .$$

NOTE: fibre and resin density should be obtained from the suppliers release documentation.

All weight measurements should be made to the nearest milligram.

1001 METHOD OF ASSESSMENT OF VOID VOLUME FRACTION OF FIBRE REINFORCED PLASTICS BY ULTRASONIC SCANNING

Introduction

This data sheet, which is an advisory document only, describes a procedure for the assessment of void content of a laminate by ultrasonic scanning technique. The procedure is valid for single through transmission using two probes, double through transmission using a plate glass reflector and double through transmission using the back surface of the laminate when it is smooth.

Equipment constraints

The following constraints with regard to equipment used for scanning of laminates is suggested.

(a) Beam angle - normality

The ultrasonic beam must be normal to the material surface within $\pm 3^{\circ}$. A reflector plate when used must be normal to the beam within $\pm \frac{1}{4}^{\circ}$.

(b) Scanning speed

Scanning speed is limited by the rate at which data can be retrieved and plotted. If the scan rate is too fast, then data plotted on a forward-running scan across an article will not match data plotted from the return run.

When an analogue signal is impressed on sensitive paper, scanning speed should not exceed 40 cm/second. Speed when using robotic scanning has been demonstrated at 65 cm/second satisfactorily but may be constrained by the speed at which the computer gathers and processes information.

(c) Noise

Ultrasonic noise is caused by water turbulence and is usually produced when water jets are used to couple the material to the transducers. Turbulence is caused primarily by air bubbles within the transducer housing and should be reduced to below ±1 dB before attempting to scan. Noise can be seen on the cathode ray tube. Electronic noise can be minimised by reducing the amplifier gain control setting.

Misalignment of the probes will also contribute. It is important to normalize each probe individually to obtain the best top surface echo before sliding the probes in-line to obtain coincidence.

(d) Probes and interface equipment

Probe performance has been shown to be age dependent. It is therefore necessary to carry out regular checks on probes to determine their useful life by monitoring their performance against a known standard.

Tests have also indicated that calibration of probes is also dependent upon the interface equipment used and therefore similar probes or interfacing equipment cannot be interchanged without re-calibrating the equipment set up.

Ultrasonic scanning of composite

Ultrasonic scanning as a means of determining the quality of composite components has been used for some considerable time usually in conjunction with a hard copy printout which shows the overall quality of the component. The copy usually in different tones between black and white or in several colours are representation of the ultrasonic attenuation of the area being scanned.

The attenuation at any position is dependent on several factors, *ie* surface texture, void, delamination and the cure reaction of the resin and fibre/resin interface. The attenuation value is also dependent on thickness of the component and therefore several density tones can be attributed to one attenuation level on a varying thickness component indicating varying levels of quality.

Of the parameters affecting the attenuation level of a composite the effect of thickness can be assumed to be linear and can be taken into account in determining quality. The cure of the resin and fibre resin interface has a minimal effect and can therefore be disregarded. Surface loss is dependent on the surface texture of the component and is independent of thickness, however errors in surface losses when applied to thin components, *ie* less than 1.5 mm can seriously affect void volume estimates.

Determination of surface losses and attenuation coefficients

A procedure for determining the above parameters is described in Ref 1. However it is recommended that the stepped laminate (Fig 1001.1) be fabricated in varying thickness and not ground from a constant thickness plate since surface losses are dependent on surface texture. Detail scan the panel to determine the lowest attenuation for each thickness using different frequency probes.

Plotting the lowest attenuation value against thickness for each frequency probe should give a graph of several linearly increasing lines (one for each

frequency probe used) originating from one point at zero thickness. This value is the value attributed to surface loss.

The slope of the lines also gives the attenuation coefficient at zero void volume in dB/mm for each frequency probe used.

Correlation of void content with attenuation

To correlate the void content against attenuation levels it is necessary to have constant thickness panels with varying attenuation level indicating therefore varying quality standards. Detail scan the panels using probes of different frequency and extract specimens 12 mm × 5 mm from areas of constant attenuation.

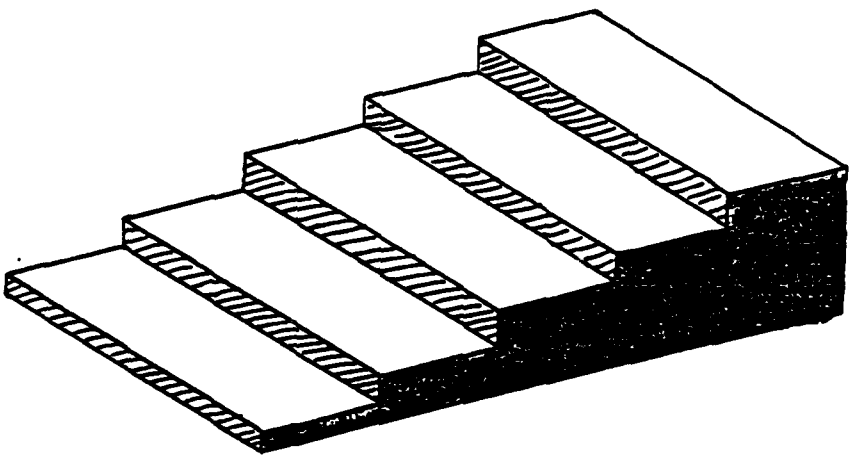
Determine the void content of the extracted specimens using the acid digestion technique as described in Method 1000.

An alternative method of correlating void content against attenuation is using through thickness examination using either Quantimet or quantitative microscopic examination (point count method). It is advisable to repeat the measurements several times polishing the specimen after each measurement to give a volumetric assessment.

A graph of the void content against attenuation level is shown in Fig 1001.2.

Reference

1 D.E.W. Store. Non-destructive determination of the void content in carbon fibre reinforced plastics by measurement of ultrasonic attenuation. RAE Technical Report 74162 (1974).



Typical thickness range: 0.75 to 6 mm

Fig 1001.1 Five step laminate

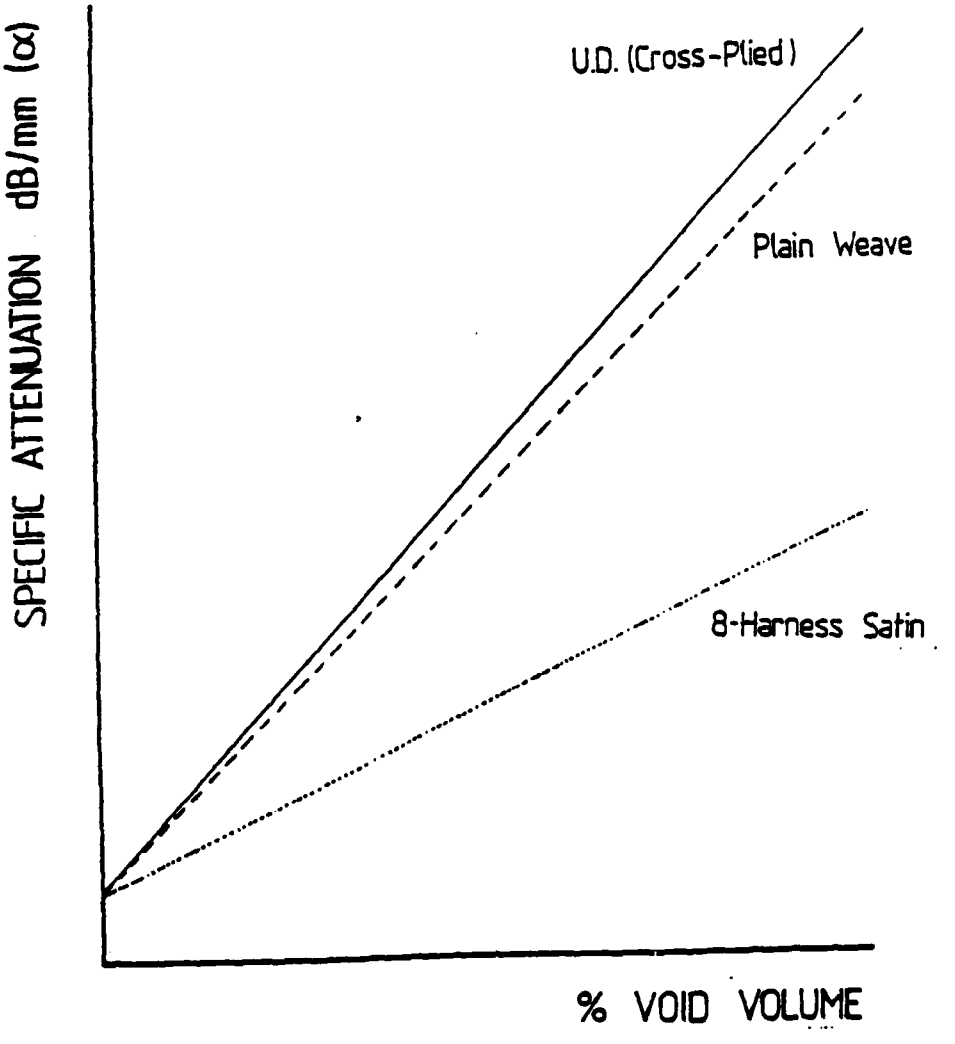


Fig 1001.2 Relationship between void volume and specific attenuation

REPORT DOCUMENTATION PAGE

Overall security classification of this page

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17. Abstract

This Report is the third and final issue of a document describing test methods suitable for the measurement of the engineering properties and other associated properties of fibre reinforced plastics. Specimen configurations and testing procedures are detailed and the applicability of the tests to the different types of fibre reinforced plastics is discussed.

F391(M)